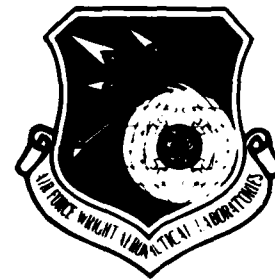AFWAL-TR-80-4156

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EVALUATION OF MICROENCAPSULATED PENETRANT INSPECTION

J.M. Portaz

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Cincinnati, Ohio   45215

December 1980

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Presented are the results of a program to evaluate the merits and short-comings of microencapsulated penetrants for use in fluorescent penetrant inspection to evaluate capsule and spray parameters to effectively use microencapsulated penetrants, and to compare the sensitivity of such a system to a conventional MIL-I-25135 Group VI liquid fluorescent penetrant inspection system. The program was divided into four phases which encompassed selection of materials for encapsulation, evaluation of capsule and spray variables, investigation of removal and developer techniques,

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measurement of sensitivity, and a process demonstration including environ-
mental and economic analysis. The work culminated with the successful
demonstration that microencapsulated penetrants could be applied in such a
way to achieve equivalent sensitivity with a MIL-I-25135 Group VI liquid
penetrant system. The microencapsulated penetrant system as now developed
uses only nonaqueous wet developers which are much more costly than dry or
aqueous wet developers. Therefore, the per-part cost of using microencap-
sulated penetrants is considerably higher than for liquid penetrants where
dry or aqueous wet developers are used.

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PREFACE

    This Final Technical Report, covering the period from 1 June 1979 to
1 May 1980, was prepared by the Aircraft Engine Quality Technology Section of
General Electric's Aircraft Engine Group (AEG), Cincinnati, Ohio, 45215,
under United States Air Force Contract No. F33615-79-C-5042. The work was ad-
ministered under the technical direction of Mr. Sidney Allinikov of the Non-
Metals Branch, System Supports Division, Air Force Materials Laboratory,
Wright Patterson Air Force Base, Ohio, 45433.

    The Program Manager is Dr. S.L. Wakefield, Manager, Materials, Composites
and Processes Technology Programs. The program was under the direction of
Mr. R.R. Wagner, Manager, Visual NDT Systems. The major contributor was
J.M. Portaz of Visual NDT Systems, Principal Investigator.

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1.0 INTRODUCTION

1.1 BACKGROUND

Fluorescent Penetrant Inspection (FPI) is one of the most widely used nondestructive inspection (NDI) methods within the Air Force Logistics Command's Air Logistics Centers and throughout the aerospace industry Many improvements in formulation of materials and in processing have been made over the years, but the basic process has remained the same. Parts are cleaned and dried, a fluorescent penetrant oil is applied and allowed to remain in the part for a specified time, the excess penetrant is removed, the part is dried, a developer is applied, and, finally, the part is inspected under black light. Proper control over each step of the process is required to obtain the most satisfactory results. Any improvement in the process that would provide for better control and/or reduced processing time while still maintaining or improving sensitivity would be of benefit to all users. The development of a powder penetrant by the Air Force Materials Laboratory represents a potential unique change in the penetrant process that has the possibility of providing such benefits.

1.1.1 Discussion of FPI Variables

There are many variables associated with the FPI process. It is important to understand the effect of each of these in order to determine the impact of a significant change in the process such as that represented by the use of a powder penetrant. In the following paragraphs each of the major process variables will be discussed relative to the potential effects that a powder penetrant was expected to create.

Part Surface Condition - As a result of the prior history of the part the surface condition will vary widely. Sand casting, for example produces rough surfaces which have the result of making excess penetrant removal quite difficult. Structures that are positioned in the gas stream of a jet engine or are exposed to high temperatures frequently have the surfaces roughened so that the same problem is encountered. In the conventional penetrant process it is necessary to control the penetrant removal very carefully so that excess penetrant is removed to the extent that nonrelevant indications due to penetrant trapped in pockets of surface roughness are kept to a minimum. At the same time the removal process must not be so aggressive that penetrant will be removed from the defects that are being sought.

A dry penetrant powder should have the advantage of not adhering to a surface simply because it is rough since there is no surface "wetting" as that which is experienced with a liquid penetrant.

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At the opposite extreme of surface conditions is the highly polished surface. This surface has the effect of making it difficult for the liquid penetrant to adhere for the full required dwell time. Thus penetrant may recede from areas that contain defects before it has time to be drawn into them and the defects will not be found. Again, since the powder penetrant does not rely on wetting and capillary action to enter a flaw it should have had potential advantages.

Part Material - In addition to the surface condition of a part the actual material from which it is made can affect the FPI system's effectiveness. The primary alloy bases used in the aircraft industry include magnesium, aluminum, iron, cobalt, nickel, and titanium. GE-AEG has performed studies to measure capability of different pentrant processes on most of these alloy bases. It has been demonstrated at a high level of statistical confidence that there is no difference in a given process' capability when used on nickel- or iron-base alloys for example. Conversely, it was also demonstrated that the identical processes were significantly less capable of detecting flaws on a titanium-base alloy. This phenomenon appears to be related in part to the wetability differences between the alloys since surface finish was identical on all test specimens.

Since the powder penetrant process would not rely on wetability to detect flaws it could have an advantage over the conventional process.

Precleaning - A key element in the FPI process that affects reliability is the precleaning of parts and their surface preparation. Methods of cleaning will be determined by the history of the part and may differ significantly between newly manufactured parts and engine run parts. Both part categories may require detergent steam cleaning, vapor degreasing, grit blasting, ultrasonic cleaning or acid etching. In addition engine run parts may also require the use of desmutters, derusters, descalers, and fluoride ion cleaning. In assessing the effectivnesss of a cleaning procedure it is important to determine that it not only provides a clean appearance but also does in fact remove interferring matter from defects. It is equally important that the cleaning method does not of itself mask over defects.

Good cleaning methods will still be required to achieve the benefits of powder penetrants.

Penetrant Dwell Time - The basic mechanism that lies behind the use of conventional FPI is the ability of a liquid to be drawn into a narrow opening through capillary action. When a flaw is very tight the rate at which a penetrant enters is apparently severely reduced. Thus lengthy dwell times are frequently required on parts that are subject to flaws of this nature. When large quantities of parts are being processed long dwell times can require special storage and handling facilities in order to provide sufficient dwell time.

The use of a powder penetrant of the correct particle size might eliminate the necessity for dwell time since a flaw opening should be completely filled immediately upon application of the powder.

Excess Penetrant Removal - After a penetrant has had sufficient time to enter a flaw the excess must be removed from the surface of a part so that the flaw alone will fluoresce during inspection and be readily detectable against a nonfluorescing background. Present liquid penetrant systems are removed by water alone if the formulation is a water washable one or by emulsification or hydrophilic removal if it is a post emulsifiable type penetrant. In either case the removal process must be performed under carefully controlled conditions to achieve complete excess penetrant removal without removing penetrant from the flaws. This requires control of several variables including emulsifier or remover concentration, the time of contact with emulsifier/remover, the temperature of the fluids, the pressure of removers and rinse water is they are sprayed on, and the length of the water rinse time.

Since the AFML feasibility studies have indicated that powder penetrant particles are firmly wedged in flaw openings but not tightly adherent to the overall surface of a part, this should facilitate removal of the excess penetrant.

Part Drying - The present conventional penetrant process requires a drying step since water is used in all mass production applications. Drying is required to remove excess moisture prior to developing if a dry powder is used or after developing if a water soluble developer is used. Studies conducted by GE-AEG have shown that both the drying temperature and time must be carefully controlled or fluorescent brightness of indications will be diminished.

A dry penetrant powder that could be removed by a dry process would eliminate the need for a drying step in the penetrant process.

Developers - Developers are employed in the conventional penetrant process to draw penetrant from indications so that they will fluoresce more brightly while still retaining their basic shape and thus be more visible to the inspector. A developer is required to improve visibility of flaws because in the conventional process some of the penetrant is usually removed from the flaw opening during the emulsification and rinsing procedures. Dry powder developers consisting of finely divided absorbent particles are used in most aerospace applications. Aqueous wet developers which are either solutions or suspensions in water of materials that are highly absorbent after drying are usually used where less sensitivity is required. A nonaqueous wet developer (NAWD) consists of highly absorbent materials suspended in an organic solvent and is used when surface roughness allows and when the highest level of sensitivity is required. Improved sensitivity is accomplished with the NAWD since the carrier fluid is an organic solvent that briefly contacts the penetrant in a flaw, mixes with it, and subsequently draws it to the developer particles on the surface. Selection of the type of developer to use depends mainly on the criticality of the part being inspected as well as the anticipated difficulty of detecting flaws which are not likely to occur on the part. The ultra high sensitivity achieved by NAWD is not always necessary when inspecting aircraft engine parts and in some cases may not be desirable due to the higher level of fluorescent background which NAWD causes on rough surfaces.

Since a penetrant powder will be held at the surface of a flaw after it is applied and will not be removed during the excess penetrant removal process there would be no need for a developer under most circumstances. However, it is possible that sensitivity of this system could be improved even further, if desired, by using a developer that would dissolve the powder penetrant capsule wall and broaden the indication to create a larger area of fluorescence and thus make it more visible.

1.2 OBJECTIVE

The purpose of this program was to evaluate the merits and shortcomings of encapsulated penetrants, (penetrant powders) with the objective of demonstrating equivalence of such a penetrant powder system to a conventional MIL-I-25135 Group VI penetrant system. The initial emphasis was directed toward the evaluation of penetrant powder process variables in order to establish a simulated production mode procedure. Later work was directed toward comparison of the penetrant powder system with a conventional penetrant system in a simulated production mode penetrant inspection line.

1.3 SCOPE

This 11 month technical effort was expected to result in improved knowledge and understanding of a powder penetrant system that had been developed by the Air Force Materials Laboratory (AFML). This improved knowledge was used to define the powder penetrant process capability and procedures for use of a powder penetrant system in a production line mode of operation. Consideration was given to the applicability of this new system to inspection of engine-used hardware as well as newly manufactured hardware.

The program consisted of four phases. The primary emphasis of Phase I was to demonstrate the sensitivity of penetrant powders using MIL-I-25135 sensitivity groups as a frame of reference. Phase II was directed toward development of powder application techniques. The emphasis of Phase III was on excess penetrant powder removal techniques. Finally, in Phase IV, the penetrant powder process was compared with a conventional penetrant process in a simulated production inspection line mode.

1.4 PROGRAM APPROACH

1.4.1 Phase I - Demonstrate Powder Penetrant Sensitivity

In Phase I, GE was involved in tasks related to the selection and screening of candidate materials for microencapsulation, preliminary production of microencapsulated materials, and finally, the measuring of the sensitivity of a microencapsulated fluorescent penetrant inspection system. In selecting candidates for encapsulation since the microencapsulated penetrants do not need to exhibit the typical characteristics associated with liquid penetrants such as wetability, primary consideration was given to brightness and ease of

encapsulation. Since it is possible that the sensitivity of the penetrant process could be further improved through the use of materials that fluoresce more brightly than currently available penetrant formulations other materials such as penetrant concentrates and single fluorescent dyes were evaluated.

Prior to actual demonstration of the sensitivity of the microencapsulated penetrants it was necessary to evaluate the effects of varying capsule parameters. To do this laboratory quantities of microencapsulated penetrants were manufactured consisting of two different penetrants, two size ranges, and two wall thicknesses.

After evaluation of application, removal and developer techniques as defined in Phases II and III of this program the sensitivity of the microencapsulated penetrant was measured. The measure of sensitivity was based on performance of the microencapsulated penetrant on 16 low cycle fatigue Inconel 718 and Titanium 6-4 test specimens supplied by the Air Force as well as on actual engine hardware with varying types of discontinuities. These results were compared with the conventional MIL-I-25135 Group VI liquid penetrant on the same test specimens in its normal mode of application.

1.4.2 Evaluate Application Techniques

The microencapsulated penetrant produced during Phase I was used for evaluation of application techniques. Due to time constrictions these techniques were limited to pressurized methods since these held the most promise for success. Variables included in the evaluation were spray distance and pressure, impingement angle, capsule size and wall thickness, spray pattern, spray time, and electrostatic versus conventional spraying.

One of the most attractive potential features of a microencapsulated penetrant system was the possibility of nearly 100% recovery and reuse of the overspray. Methods of recovering the overspray were investigated concurrently with the task to evaluate application techniques.

As a result of the application technique evaluation and the sensitivity evaluation a decision was made to produce a particular type and size of microencapsulated penetrants. A pilot lot of microencapsulated penetrants was produced both as a test of the encapsulation process to produce a consistent material and to provide sufficient material for later production mode line testing. This was made to a specification based on the results of Phase I evaluation and preliminary production experience.

1.4.3 Evaluate Removal and Developer Techniques

Although the microencapsulated penetrant system essentially uses a dry material some particles do adhere to the surface of test arts and create an objectionable background fluorescence. This adherence is due to various factors including capsules bursting and releasing oil, compacting of the

particles and electrostatic forces. This excess fluorescent material must be removed in order to produce an acceptable surface for inspection. In Phase III of the program, GE performed an evaluation of various removal techniques including high pressure air, solvent dipping and brushing, and detergent dipping and brushing.

Developers are used in conventional systems to draw out penetrant from flaws and broaden the indications so that they are more readily detectable by an inspector. Microencapsulated penetrants were not expected to respond to developers in the usual mode of application since no fluid is present. Developing methods concentrated on ways of drawing the liquid penetrant from the capsules.

1.4.4 Process Demonstration

Phase IV included the demonstration of both conventional liquid and microencapsulated penetrant processes in a simulated production mode. The inspection rate capability, plus the estimated cost of inspection per part, was determined. Environmental and health consequences of the microencapsulated penetrant technology were assessed from the standpoint of the project itself and of future scale up to production quantities.

2.0 DEMONSTRATE SENSITIVITY

2.1 SELECT CANDIDATE MATERIALS FOR ENCAPSULATION

2.1.1 Introduction

The first major task of the program was to obtain samples of liquid fluorescent penetrants and other fluorescent materials to use as candidates for encapsulation. It was anticipated that the penetrant that was encapsulated would also be used in its conventional liquid form for comparison of sensitivity and in the production line comparison. A primary criterion for selection was therefore a combination of high fluorescent brightness and excellent removability. However, since conventional removability would not be a factor in its use as an encapsulated material, brightness was an overriding factor. Further, the encapsulating process itself is facilitated if the material contains little or no surfactants.

With the above mentioned criteria in mind, each of the major penetrant manufacturers who serve both the domestic and foreign aircraft industry were contacted for recommended candidate materials. These manufacturers included Uresco, Met-L-Chek, Brent Chemicals (Ardrox), Magnaflux, Turco, Sherwin, and Oxy Metal Industries (OMI). Discussions were held with knowledgeable personnel of these companies and candidate materials were selected for preliminary brightness and encapsulation tests. Of the seven manufacturers that were contacted all but one responded with samples of penetrant material. Penetrants received were Sherwin Products' RC77, Ardrox's 985P3, Turco Products' P60B, Oxy Metal Industries' P6F-4, Met-L-Chek's FP-97, and Magnaflux's ZL22C and ZL30A.

The purpose of selecting additional materials is to explore the possibility that a brighter penetrant, and therefore a more "seeable" penetrant, might be manufactured in the encapsulated form than is possible in the conventional liquid form. A major problem in increasing the brightness of conventional penetrants is the inability to keep high concentrations of fluorescent dye in solution while maintaining wetability and removeability characteristics. Since wetability and removeability should not be factors in the use of encapsulated penetrant the usual constraints on dye concentration would not be felt. At the same time that penetrant suppliers were contacted for candidate conventional materials, the subject of concentrates was also reviewed. Response to this request was much less enthusiastic. Only three manufacturers supplied samples. Materials received were Sherwin Products' LAB-B913 concentrate, Met-L-Chek's FP-97 concentrate, and Brent Chemicals Corporation LT79/9/144. However, this last sample was received too late and in too small a quantity for original brightness measurements and encapsulation.

2.1.2 Encapsulating Ability

All initial work on developing a technique for encapsulating fluorescent penetrants as well as all of the preliminary laboratory production of encapsulated penetrants was performed by Capsulated Systems, Inc., Yellow Springs, Ohio. This work was initiated and supported by the United States Air Force Materials Laboratory. Approximately half way through the program the Air Force program manager, S. Allinikov, identified a second vendor (Djinni Industries, Dayton, Ohio) whom they felt could produce encapsulated penetrants of sufficient quality for our purpose.

A key factor in determining which penetrants to use for encapsulation is the ease of encapsulation. It was known from discussions with Capsulated Systems that the ease with which a material encapsulates is dependent on its composition. The following fluorescent materials were submitted to Capsulated Systems, Inc. for encapsulation.

BIO-PEN-P6F-4
Zyglo ZL-22C and ZL30A
Dubl-Chek HM-607 and RC-77
Dubl-Chek Lab. B913 (concentrate)
Ardrox 985P3
Met-L-Chek FP-97 and FP-97C (concentrate)
Fluro-Check P60B

Of these, only Zyglo ZL-30A and ZL22C, Met-L-Chek FP-97, and Dubl-Chek RC-77 were encapsulated successfully.

In the encapsulation process an emulsion is first made whereby the penetrant is suspended in droplets of the required diameter. The encapsulation media is then introduced and forms a covering around the droplets. The problem with the materials that did not encapsulate successfully was that the droplets formed were either too large, or if small droplets did form, they would quickly coalesce to form larger droplets. Capsulated Systems proposed that with additional experimenting these materials could be successfully encapsulated; however, the cost would be greater. Due to constraints of program timing and cost this was not pursued any further.

Approximately half way through the program Djinni Industries did encapsulate a single fluorescent dye from Da-Glo. Although it was not in sufficient quantity to spray and was too late in the program to effectively evaluate, the fact that materials other than penetrants can be encapsulated leads to the possible formulation of much cheaper, brighter, and versatile fluorescent penetrants.

2.1.3 Brightness Measurements

An important factor in selecting a Group VI penetrant for encapsulating was a high fluorescent brightness. Since the penetrant is to be encapsulated other factors such as removability and wetability were not considered meaningful.

Using an IL700/760/780 Spectroradiometer System from International Light the intensity of light emitted by the penetrants was measured at wavelengths from 2500Å to 8000Å in 100Å increments. This range includes the upper ultraviolet, the visual and the near infrared regions. The test specimen was a 1.5-inch diameter shallow container with approximately a 1/8 inch layer of encapsulated material completely covering the bottom. Ultra-violet lamp output measured at test specimen with a J221 black ray meter was held constant at 1000 microwatts per square centimeter. Distance from speci-men to lens was six inches. A setup of the system is illustrated in Figure 1. Results for each penetrant successfully encapsulated are illustrated in Figures 2 through 7. It is interesting to note the similarity of spectral distribution for all the encapsulated materials. This may imply that all major penetrant manufacturers use the same basic dye or combination of dyes. The reason for this may be that this dye or dyes provide adequate brightness and have the capability to be easily put into a solution containing the required characteristics of a fluorescent penetrant, i.e., wetability, vis-cosity, surface tension, removability, etc. Since these restraints do not apply to encapsulated penetrants, there is the possibility of encapsulating a dye that has superior brightness but which lacks other requirements necessary in liquid penetrants. The table below lists the maximum brightness in the visual range of each penetrant after encapsulation.

ZL-30A (Batch 80-9)	675×10^{-10} W/cm^2
ZL-30A (Batch 80-25)	460×10^{-10} W/cm^2
ZL-22C (Batch 80-27)	584×10^{-10} W/cm^2
RC-77 (Batch 80-26)	323×10^{-10} W/cm^2
FP-97 (Batch 80-21)	317×10^{-10} W/cm^2
FP-97 (Batch 80-28)	298×10^{-10} W/cm^2

The difference between batches 80-9 and 80-25 of the ZL-30A and 80-21 and 80-28 of the FP-97 is that batches 80-25 and 80-21 were encapsulated by a different process which would make the capsules more impervious. However, in the case of the ZL-30A it also significantly reduced the brightness of the penetrant.

Spectral distributions of liquid unencapsulated ZL30A is shown in Figure 8. As can be seen, the response of the liquid material is similar to that of the encapsulated material in all regions except this infrared where the encap-sulated material peaked at 7600Å and then declined in brightness. Spec-tral distribution of all other liquid materials tested was similar to that of ZL30A with the only difference being the brightness magnitude. There is a significant drop in the brightness of the encapsulated materials when compared with the unencapsulated materials; however, no attempt should be made to di-rectly compare results since two different methods were required to hold the liquid and encapsulated materials. The test specimen for the liquid material
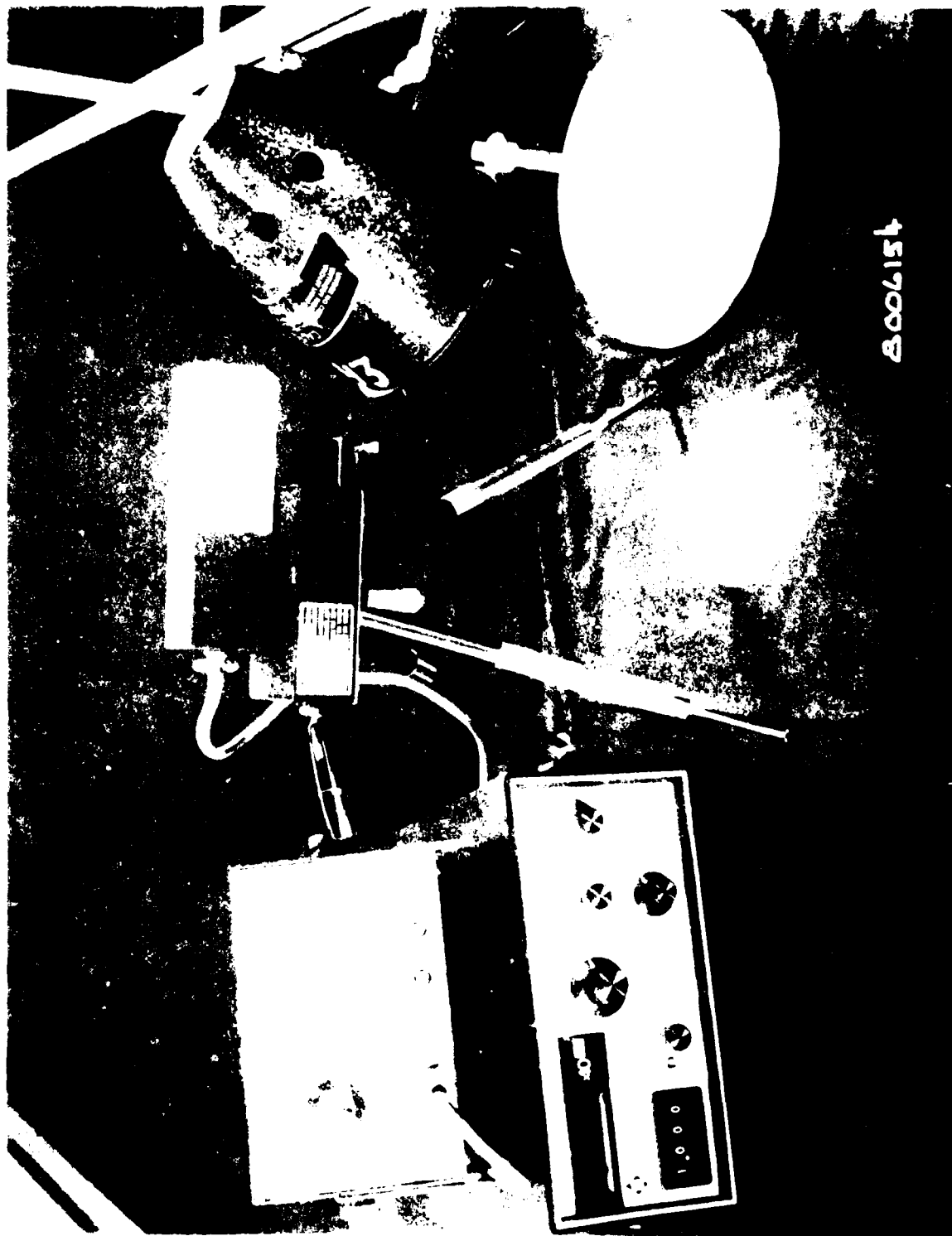
Figure 1 — Spectroradiometer System

Figure 2. Encapsulated ZL30A (Batch 80-9) Spectral Pattern.
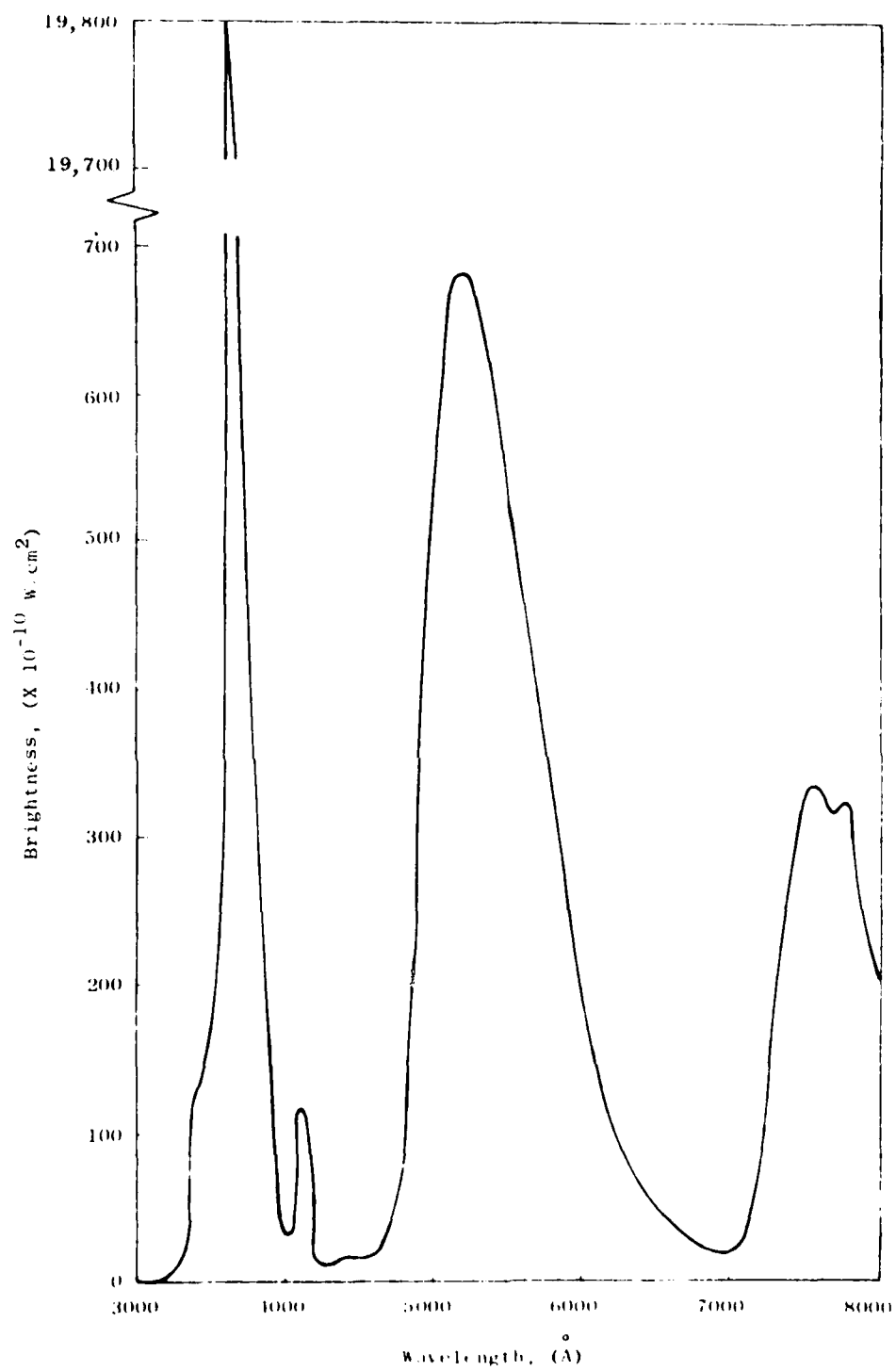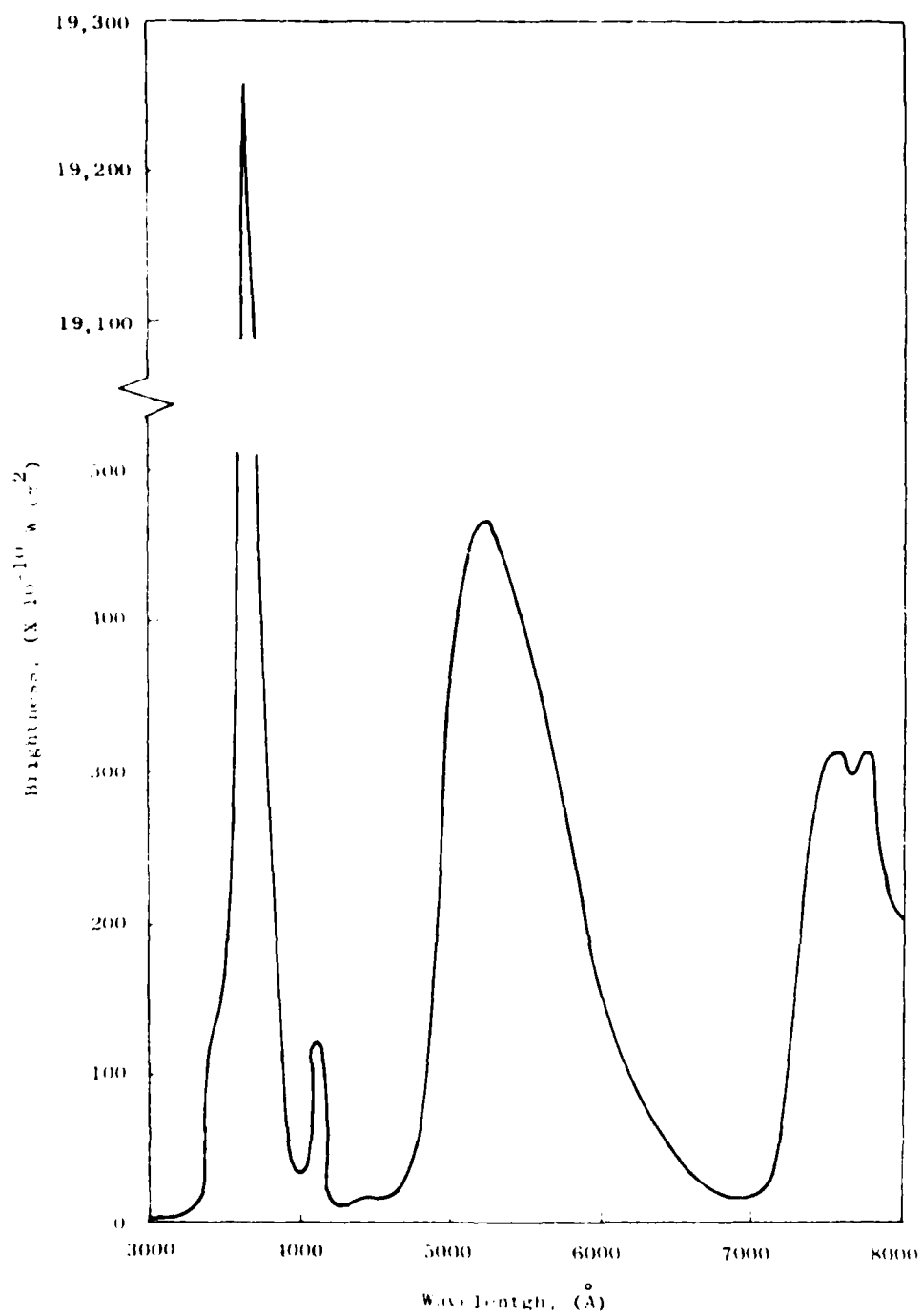
11

Figure 3. Encapsulated ZL30A (Batch 80-25) Spectral Pattern.
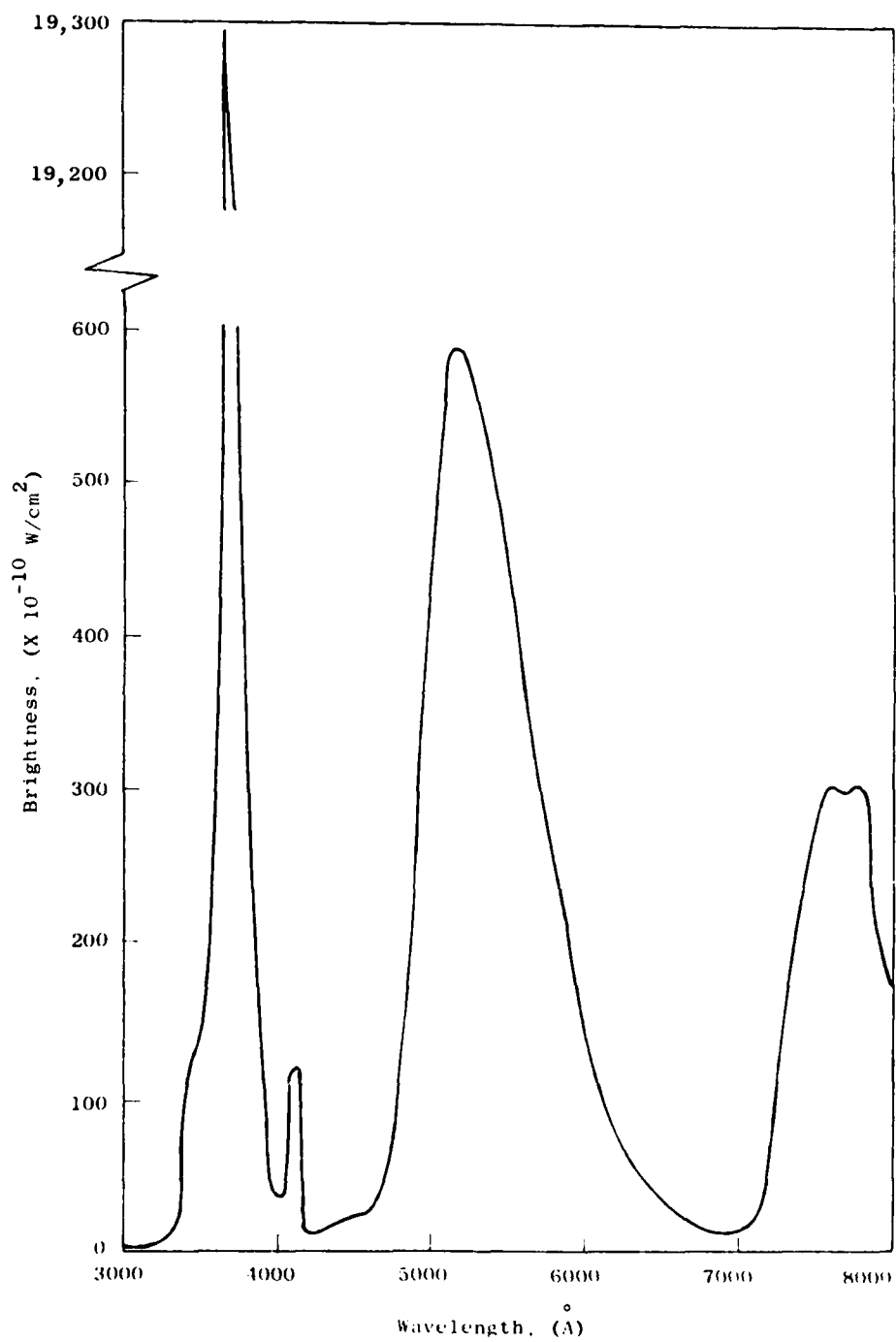
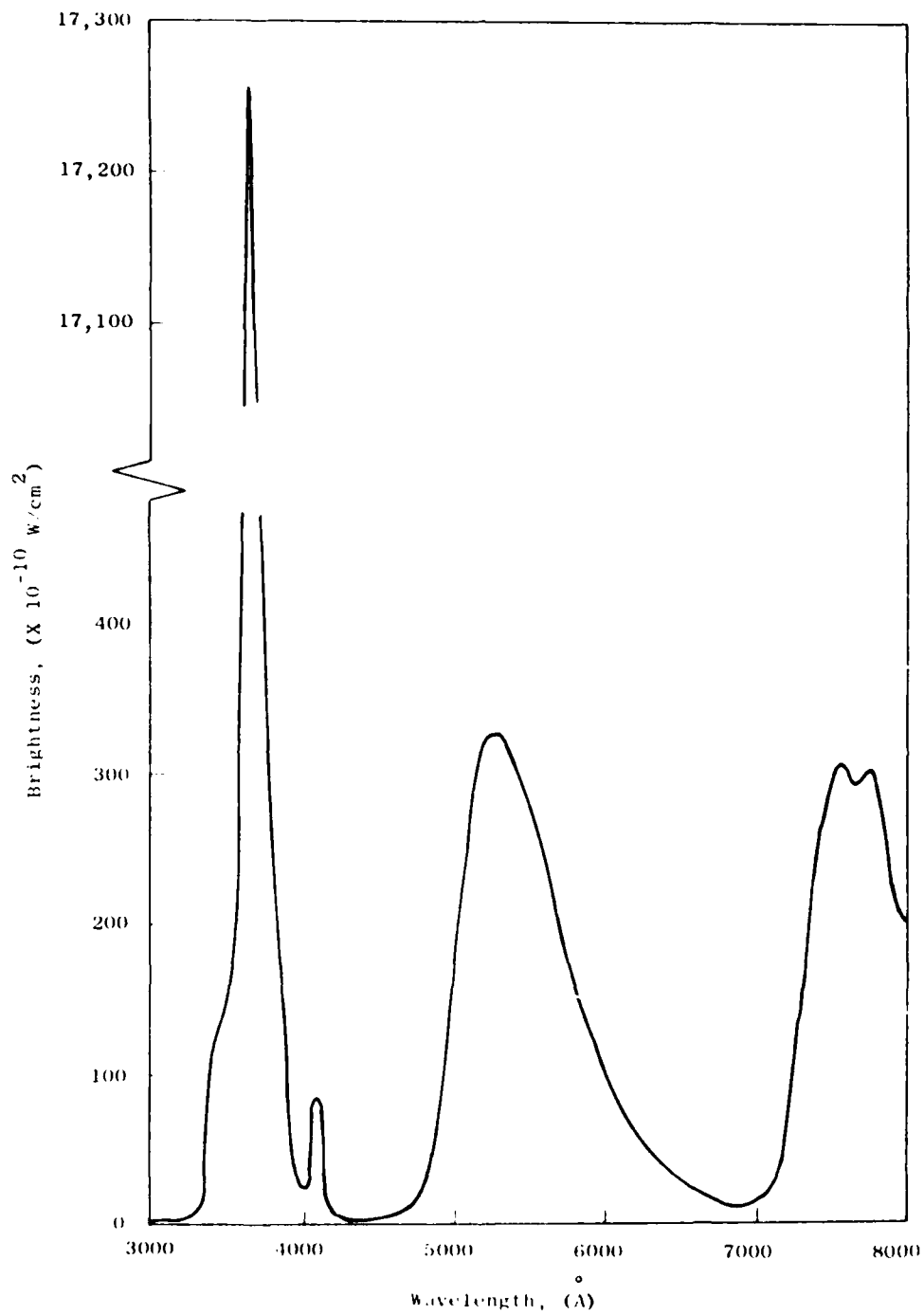Figure 4. Encapsulated ZL22C (Batch 80-27) Spectral Pattern.

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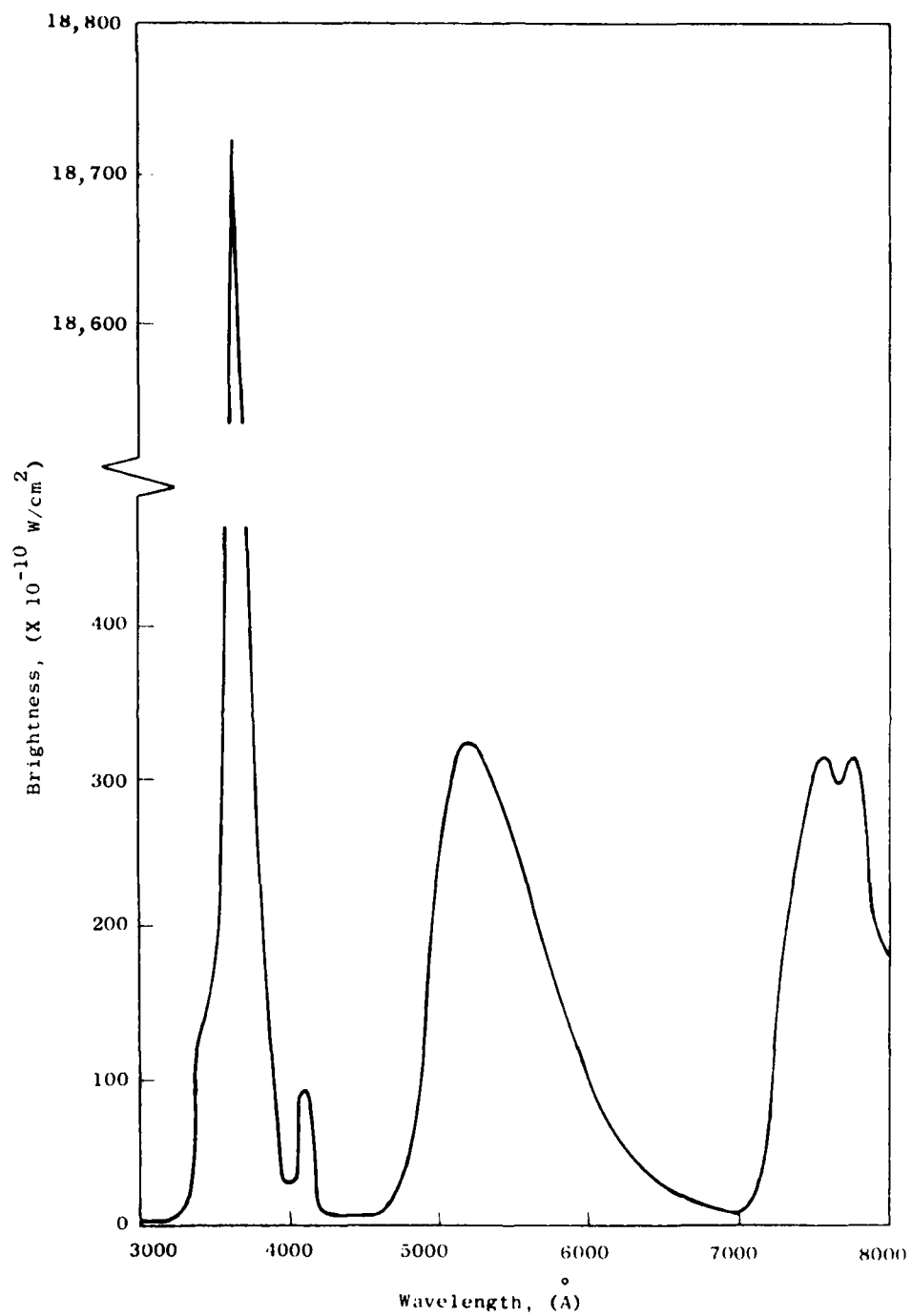Figure 5. Encapsulated RC-77 (Batch 80-26) Spectral Pattern.

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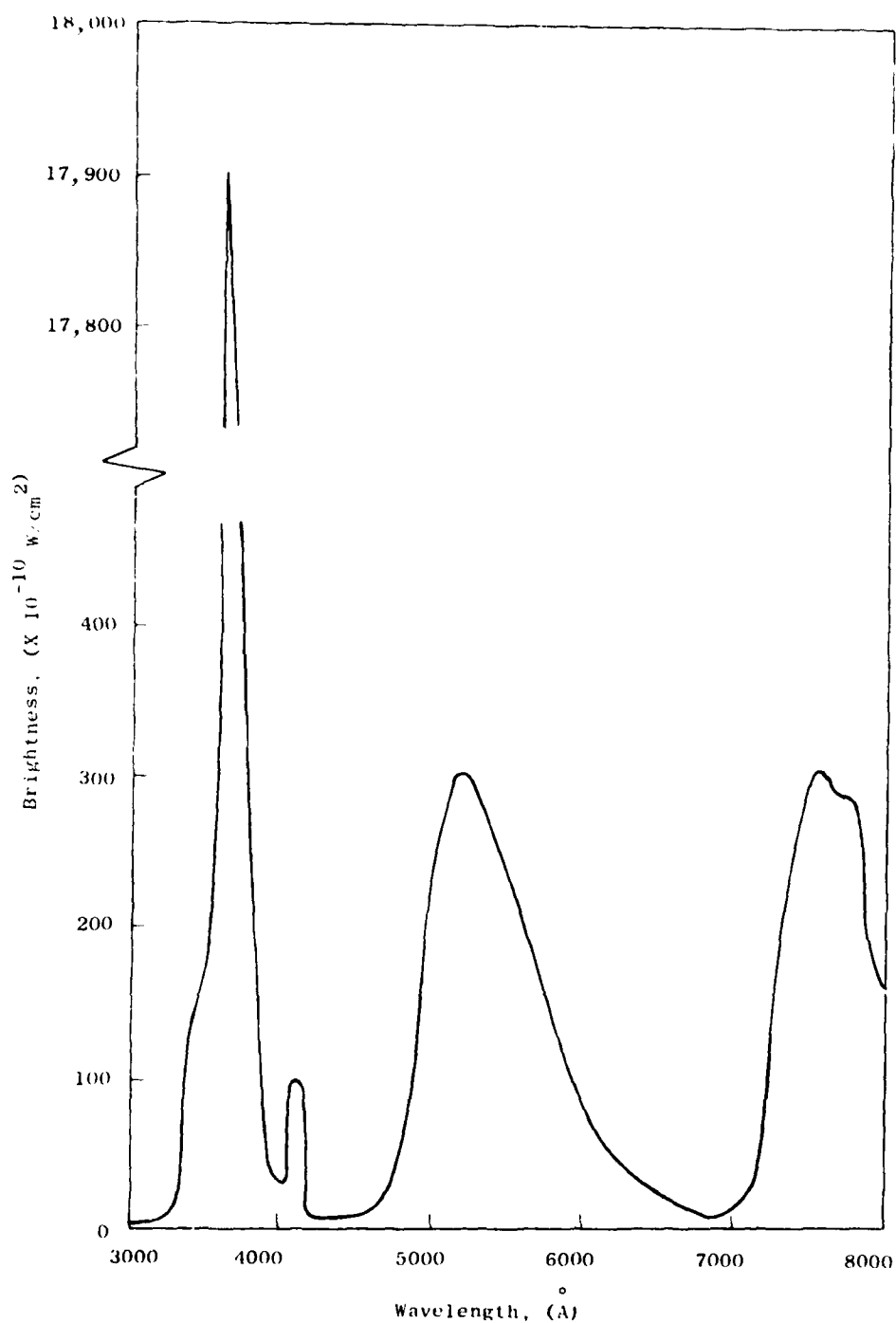Figure 6.   Encapsulated FP-97 (Batch 80-21) Spectral Pattern.

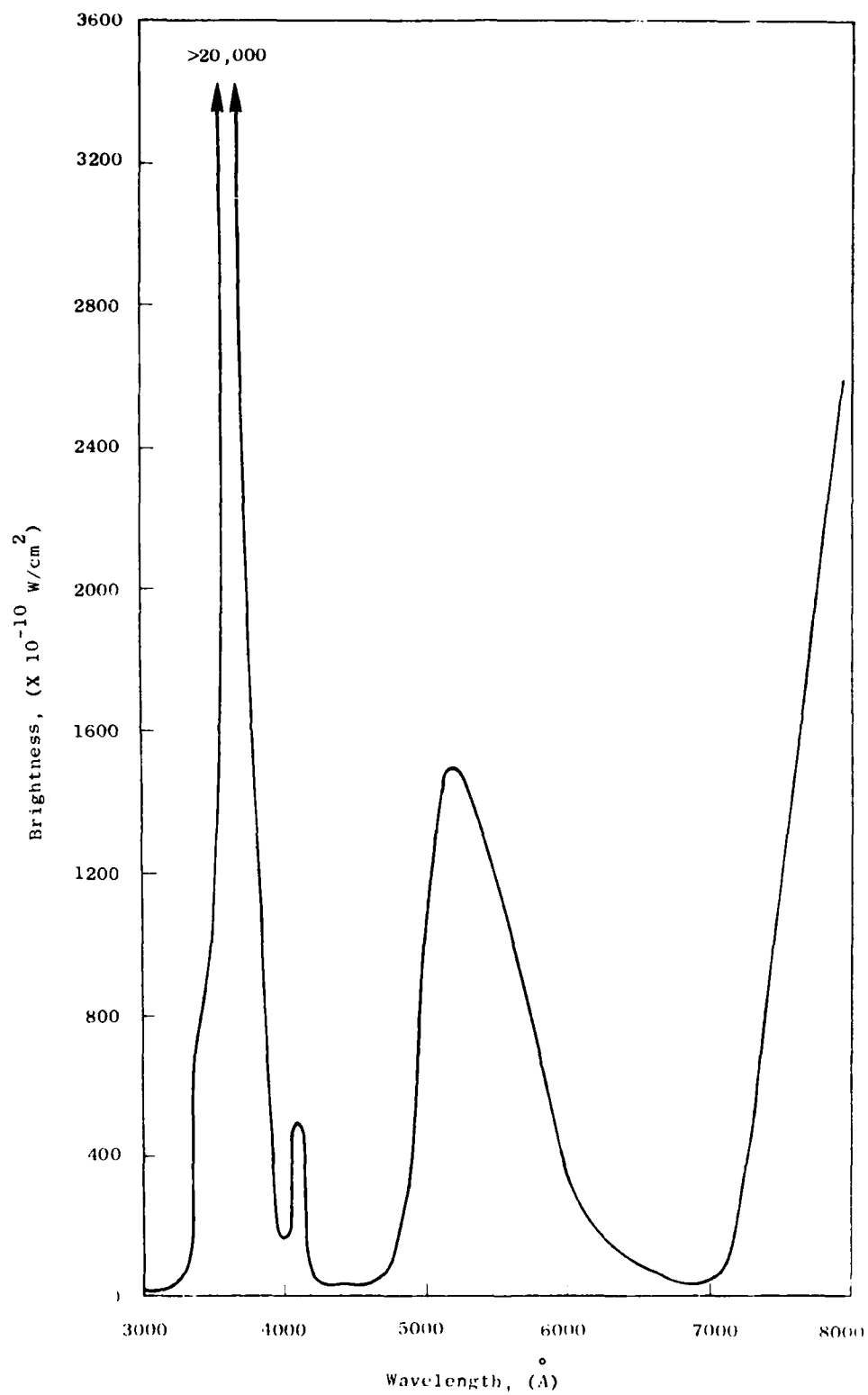Figure 7. Encapsulated FP-97 (Batch 80-28) Spectral Pattern.

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Figure 8. Liquid ZL30A Spectral Pattern.

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was a 5.5 cm filter paper with four drops of penetrant that were allowed to
spread evenly. That for the encapsulated material has been previously de-
scribed.

2.2 PRELIMINARY PRODUCTION

Based on encapsulating ability and brightness two penetrants were se-
lected for further evaluation. These were ZL-30A and RC-77. ZL-30A dis-
played the best brightness characteristics of the penetrants encapsulated and
was chosen for that reason. There was a difference in the manner that ZL-30A
and ZL-22C versus RC-77 and FP-97 were encapsulated. RC-77 and FP-97 micro-
encapsulated as single droplets, 1-5 microns, with little aggregation. Pene-
trants ZL-30A and ZL-22C formed small, loose aggregates, 2-8 microns in size,
after microencapsulation. Per Capsulated Systems, Inc., the flowability of
both ZL-30A and ZL-22C was much better than either RC-77 or FP-97. Thus, the
selection of ZL-30A and RC-77 provided an opportunity to look at and evaluate
the effects of brightness and flowability on the sensitivity of microencapsu-
lated penetrants.

To evaluate the effect of capsule size on sensitivity, the two penetrants
were requested to be encapsulated in a diameter range of 2-5 microns and 20-40
microns. Two microns is the smallest that the penetrants could be encapsu-
lated without having to be suspended in a water vehicle. Twenty to 40 microns
was estimated to be sufficiently larger to provide a good evaluation of the
effect of capsule size. The range of capsule diameters actually received from
capsulated systems was 1 to 4 microns with three microns being the average
for the small diameter capsules and 5 to 35 microns with 20 microns being the
average for the large diameter capsules.

Capsule wall thickness was expected to have a significant effect on sen-
sitivity of encapsulated penetrants. Thin-wall capsules although more prone
to breakage should also be more flexible, allowing them to penetrate cracks
smaller than the diameter of the capsules. Thick-wall capsules although less
flexible would also be less prone to rupture. To evaluate the effect of cap-
sule wall thickness each of the large and small diameter capsules were manu-
factured with a thin and thick wall. The size of the wall is determined by
the diameter of the capsule, thus, a thick wall for a small diameter capsule
would be significantly smaller than for a large diameter capsule. The 2 to 4
micron capsules manufactured by Capsulated Systems had a thin wall of 0.074
microns and a thick wall of 0.09 microns; the 0 to 40 micron capsules had a
thin wall of 0.46 microns and a thick wall of 0.61 microns. Diameter of cap-
sules was measured by the vendor using a microscope in conjunction with a
grid. Capsule wall thickness is basically a calculated value and is dependent
on the capsule diameter and the ratio of core material to wall material. The
equation for calculating wall thickness is

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$$T = B - A = B\left(1 - \left(\frac{1}{(P_R^{-1} + 1)^{1/3}}\right)\right)$$

Where B = Capsule radius
A = Core radius
P_R = Volume ratio of core to wall

2.3 SENSITIVITY MEASUREMENTS

2.3.1 Introduction

Following the manufacture of laboratory quantities of encapsulated pene-
trants, methods of applying, recovering, removing, and developing the encap-
sulated penetrants were evaluated. These will be described in sections three
and four. This led to the determination of the effect of process variables
on the sensitivity of an encapsulated penetrant system. Knowing these ef-
fects it was possible to measure and compare the sensitivity of an encasulated
lated penetrant system with a conventional liquid penetrant system. To de-
termine repeatability of the process each test was conducted three times.

2.3.2 Selection of Test Specimens

Specimens used in measuring the sensitivity of the processes consisted
of the engine run and new production airfoils which were used throughout the
program for evaluating application and removal techniques. Also used to de-
termine sensitivity of encapsulated penetrants were the 16 low cycle fatigue
test specimens that the United States Air Force Materials Laboratory uses to
qualify MIL-I-25135 Group VIA and VIB penetrant systems. Eleven of these
blocks are Inconel 718 and five are Titanium 6-4. All blocks have only one
crack except for serial numbers 99 which has two and 94 which has three.
Crack size ranges from approximately 0.010 inch to 0.100 inch.

In selecting the airfoil specimens for use in the program consideration
was given to the following factors: material, engine type, size of defect,
type of defect, surface condition, and manufacturing processes. Candidate
parts were fluorescent penetrant inspected via the conventional liquid method.
Fifteen specimens were selected for use in the program. These consisted of
the following:

    TF34 Compressor blade rough forgings (2)
    TF34 Compressor blades finish machined (2)
    TF34 Stage 2 High Pressure Turbine blade - Engine Run
    J79 Stage 1 Turbine blade
    J79 Stage 2 Turbine blade
    TF39 Compressor vane
    TF39 Compressor blade
    TF39 Stage 2 High Pressure Turbine blade
    TF39 Stage 1 High Pressure Turbine blade (2) - Engine Run

F101 Compressor blade
Fine cracked chrome panel -F97424
Coarse cracked chrome panel -C123

Materials represented by the above hardware include nickel-base cast-
ings, and titanium and Inconel forgings. Indications included grinding
cracks, fatigue cracks, microshrinkage, forging laps, and cold shuts. Size
of indications ranged from 0.010 inch to 1.00 inch.

2.3.3 Systems Compared

In qualifying a fluorescent penetrant system the candidate materials
must be tested back-to-back, using the same specimens, with a baseline
penetrant system which has already been qualified for that particular group.
Since ZL30A was used as an encapsulated penetrant and since ZL30A had already
been qualified as a MIL-I-25135 Group VIB penetrant, it was decided to use
this as the baseline system. All parts were inspected three separate times
using ZL30A penetrant with a dwell time of 30 minutes, a one minute prerinse
with 60° F water at 20 psi, and a 30 second agitated dip in 5% solution of
ZR10A remover. Parts were then post-rinsed using 70° F water at 40 psi and
dried in a 150° F oven for approximately 15 minutes. D499C developer was
then applied and parts inspected following a seven minute dwell time. To
assure all oil from discontinuities was removed after each inspection the
parts were placed in an ultrasonic cleaning tank containing 1-1-1 trichloro-
ethane for a minimum of one and one-half hours. Ultrasonic agitation was
used for 45 minutes of this total time.

Based on results from Phases II and III of the program where application,
removal and developer techniques were evaluated the following parameters were
used to measure the sensitivity of encapsulated penetrants: encapsulated
ZL30A penetrant, 20-40 micron diameter, thick wall, 80 psi spray pressure,
two inches from spray gun to part, direct spray, and a wide spray pattern.
Excess penetrant was removed from the surface of the part by wiping with a
mild detergent solution and rinsing with 70° F water at 40 psi. Parts were
then dried in a 150° F oven for approximately 15 minutes. D499C developer
was then applied and parts inspected. A DeVilbiss MBC spray gun with a MBC-
496-C fluid needle and an AV-641-AC air cap was used to spray the encapsulated
penetrant on the parts. To assure that all capsules were removed from the
discontinuities the parts were submerged in a bath of methyl alcohol for ap-
proximately one hour. Methyl alcohol breaks down the capsule walls making
penetrant removal possible. To further assure that no foreign substance was
left in the discontinuities the specimens were then ultrasonically cleaned
with 1-1-1 trichloroethane for one-half hour.

2.3.4 Statistical Results

Tables 1 and 2 summarize the data for both the low cycle fatigue blocks
and the airfoil specimens. Photographs of indications found by the encapsu-
lated penetrant method are included in Figures 9 through 39.

In the low cycle fatigue blocks, the conventional ZL30A liquid penetrant process found a total of 55 out of a possible 57 finds and the encapsulated ZL30A penetrant found 56. Both misses by the liquid process and the one miss by the encapsulated penetrant process were of the same defect. When this defect was found it only measured a length of 0.010 inch. With the airfoil test specimens, the liquid penetrant process found 58 and the encapsulated penetrant 59 cracks. This difference of one missed indication in each instance is not statistically significant and, thus, based on crack detection there is no difference between liquid and capsulated penetrant sensitivity.

Table 1. Indication Size of Liquid Versus Powder Penetrants.

BLOCK S/N	Material	BASELINE			POWDER PENETRANT		
		ZL30A Run #1	ZR10A Run #2	D499C Run #3	ZL30A Run #1	20-40 Run #2	Thick Wall Run #3
73	Inco 718	.090	.090	.080	.090	.080	.080
6	Inco 718	.030	.035	.030	.030	.030	.030
93	Inco 718	.080	.075	.075	.080	.075	.080
61	Inco 718	.070	.065	.060	.060	.060	.060
19	Inco 718	.045	.045	.045	.045	.045	.045
86	Inco 718	.090	.090	.090	.090	.090	.090
77	Inco 718	.075	.090	.080	.080	.080	.085
67	Inco 718	.085	.090	.090	.080	.090	.090
13	Inco 718	.015	.015	.015	.015	.015	.015
83	Inco 718	.075	.080	.075	.075	.075	.075
37	Inco 718	.045	.045	.045	.045	.045	.045
83	Ti 6-4	.075	.070	.075	.075	.075	.075
45	Ti 6-4	.030	.030	.030	.030	.030	.030
17	Ti 6-4	.015	.015	.015	.020	.020	.020
99	Ti 6-4	.030	.030	.030	.030	.030	.030
		.030	.030	.030	.030	.030	.030
94	Ti 6-4	.045	.030	.050	.050	.050	.050
		.015	.015	.015	.015	.015	.015
		.010	–	–	.010	.010	–

Table 2. Indication Size of Liquid Versus Powder Penetrants.

PART	BASELINE		POWDER PENETRANT	
S/N	Run #1	Run #2	Run #1	Run #2
4	.030	.030	.030	.030
5	.020	.020	.020	.020
1	.300	.300	.30	.28
3	.030	.020	.020	.020
	.045	.045	.045	.045
12	1.00	1.00	1.00	1.00
	.65	.65	.70	.70
10	.30	.30	.30	.30
14	.40	.40	.40	.40
	.40	.40	.40	.40
15	.20	.20	.20	.20
	.045	.045	.060	.045
	–	.045	.045	.030
	.24	.25	.26	.26
	.47	.44	.45	.46
	.35	.30	.30	.32
	.20	.20	.20	.20
16	.060	.060	.045	.045
	.020	.020	.030	.030
	.030	.030	.020	.020
	.015	.010	.010	.010
	.045	.045	.030	.030
17	.030	.030	.030	.030
	.045	.045	.045	.045
	.020	.020	.020	.020
	.030	.030	.030	.030
	.220	.220	.220	.220
7	.80	.80	.80	.80
18	.250	.250	.250	.250

22

13
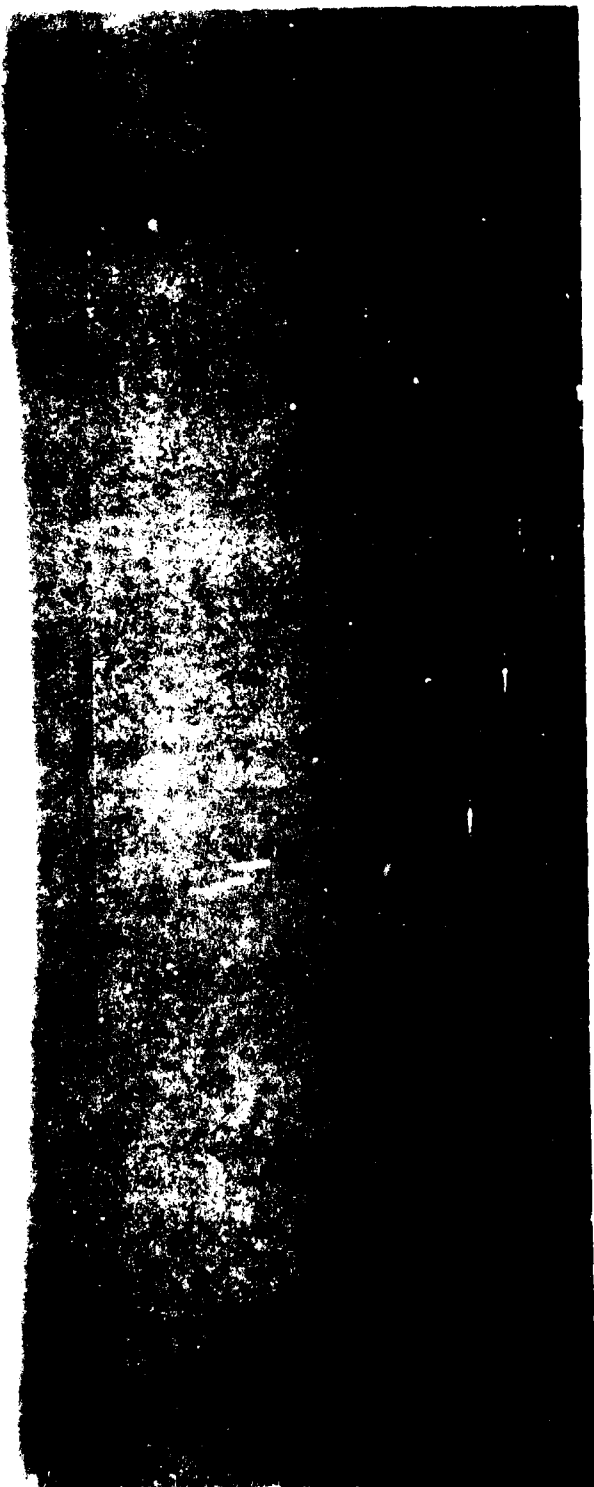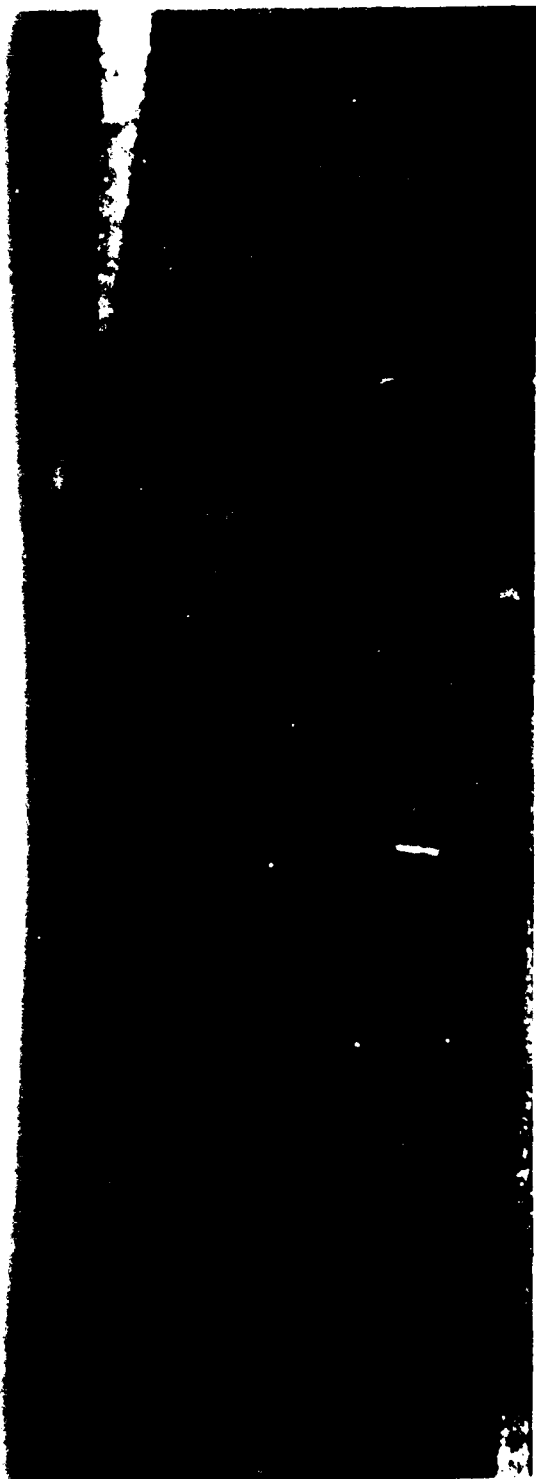
88

83T
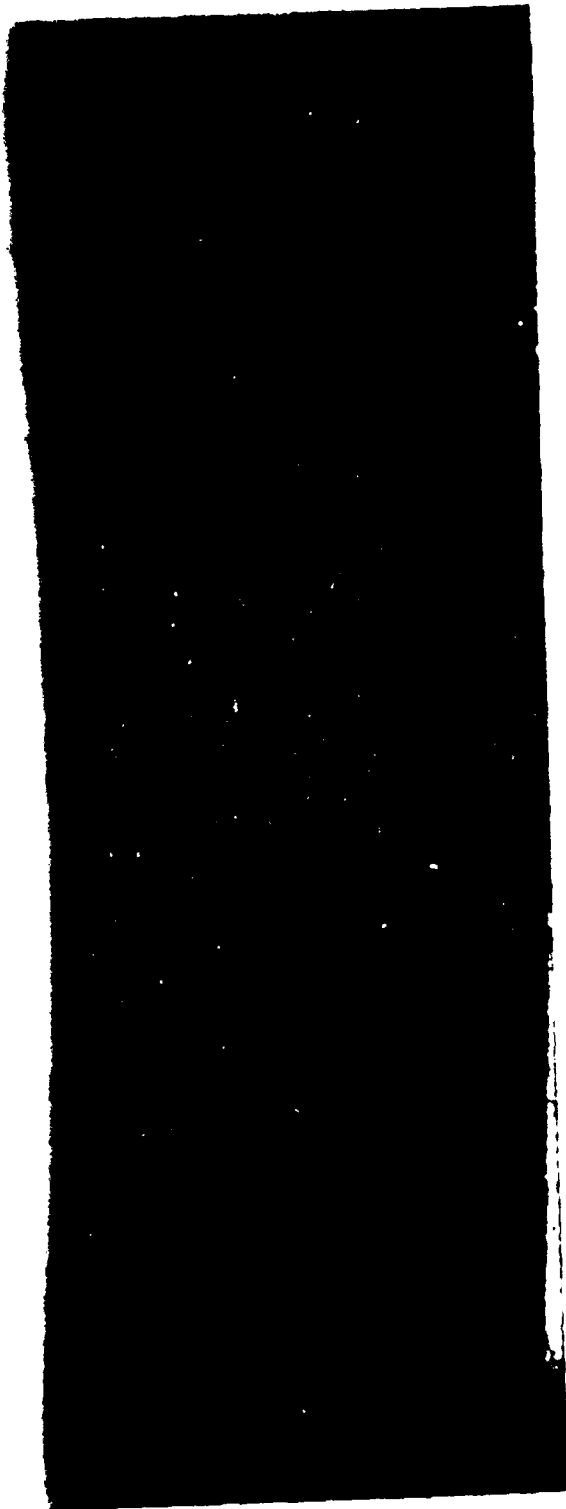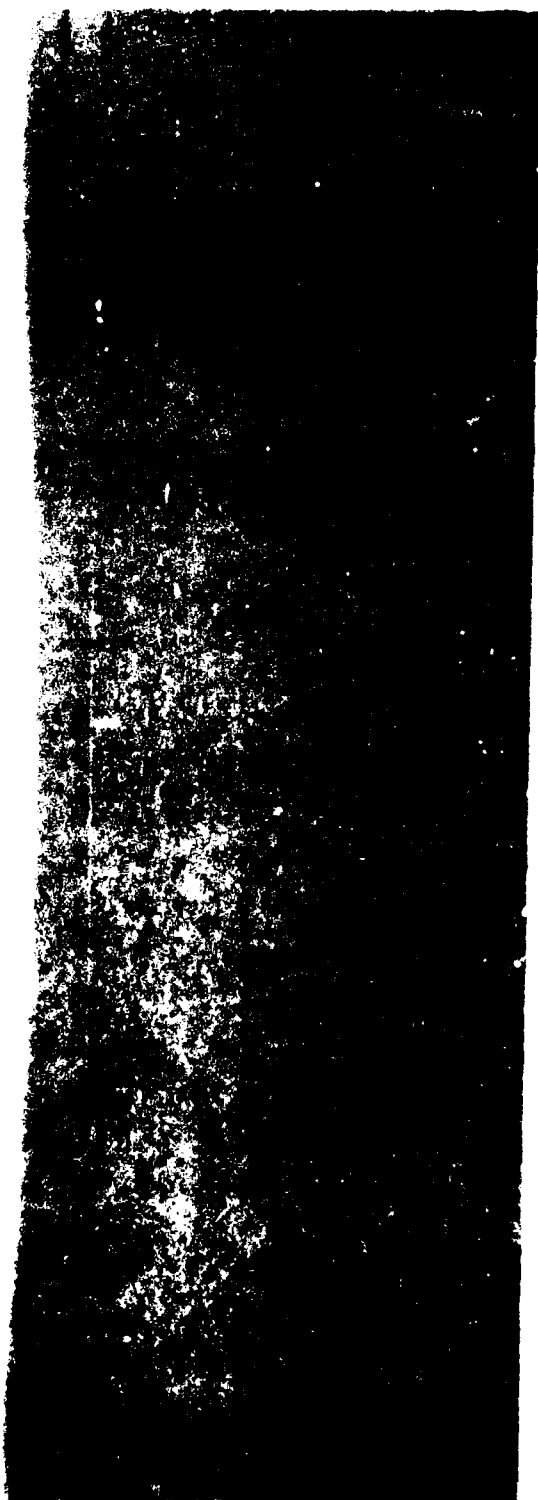
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13

94

19

45

99

11
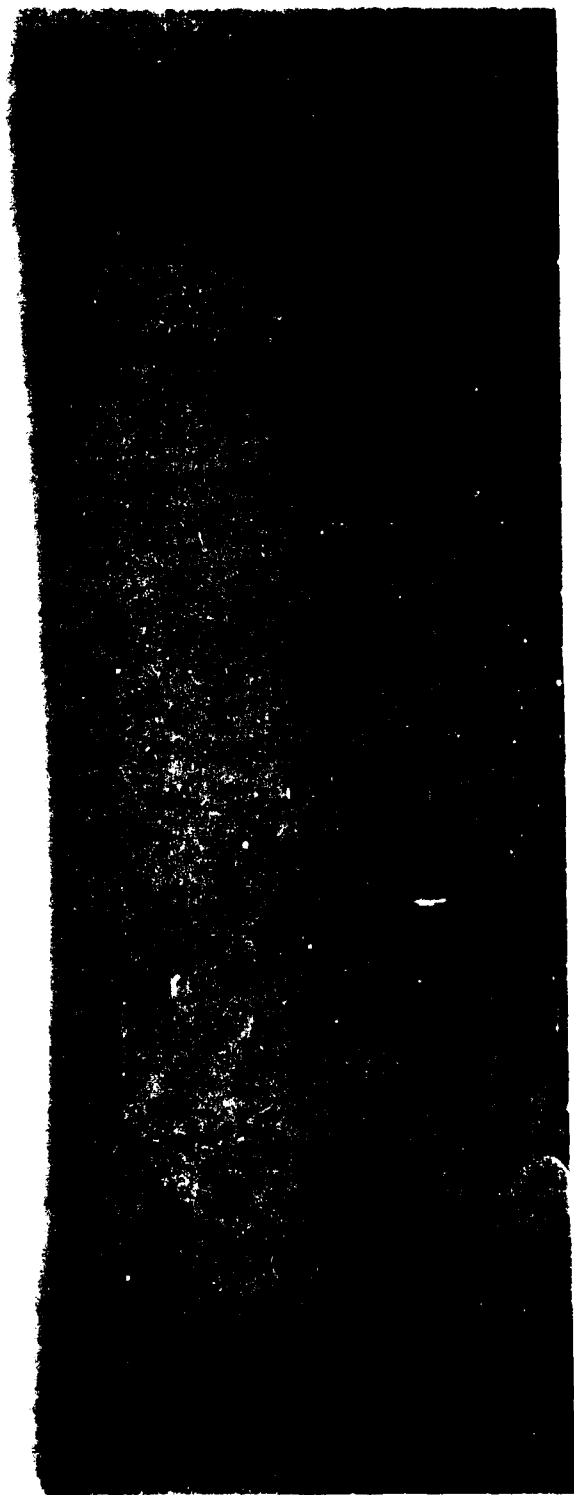
831

19

37

6
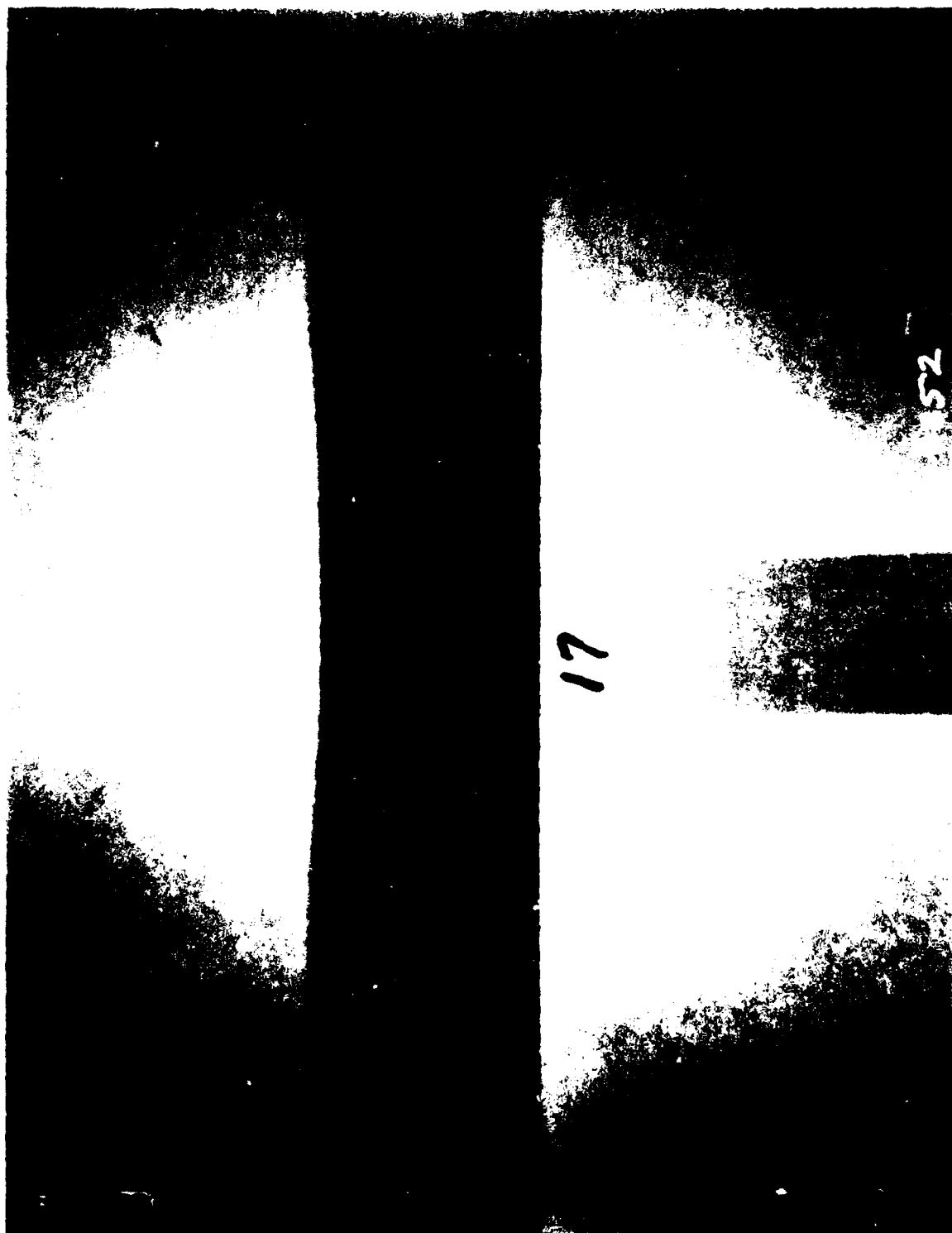
Figure 26. TF34 Compressor Blade Forging
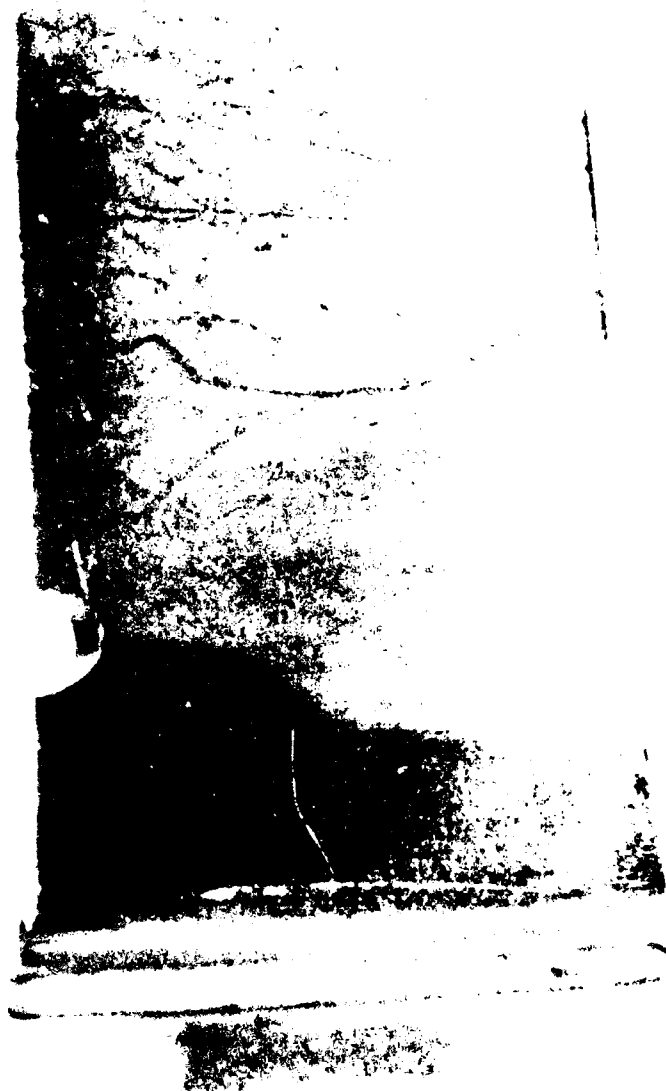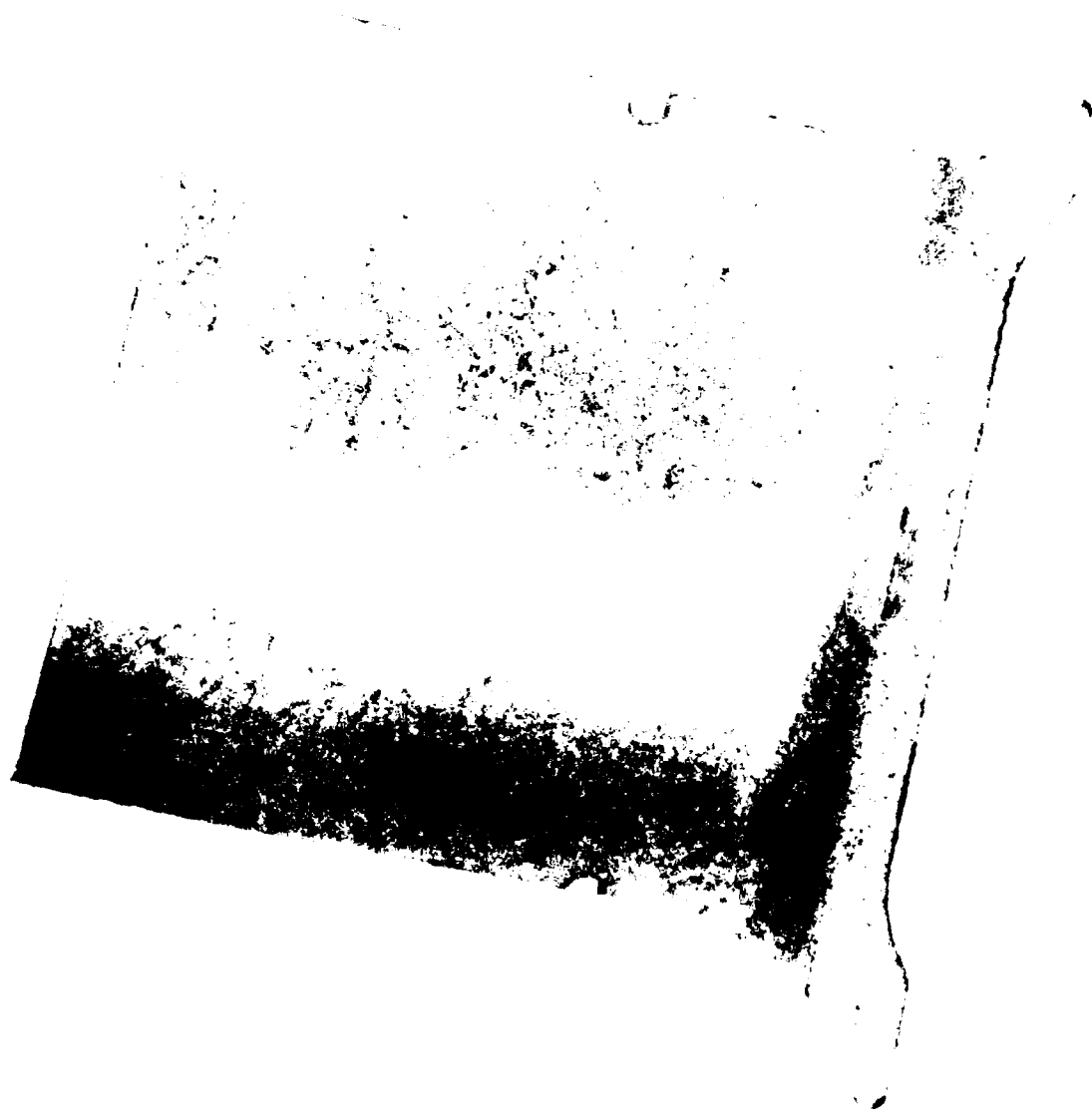
40

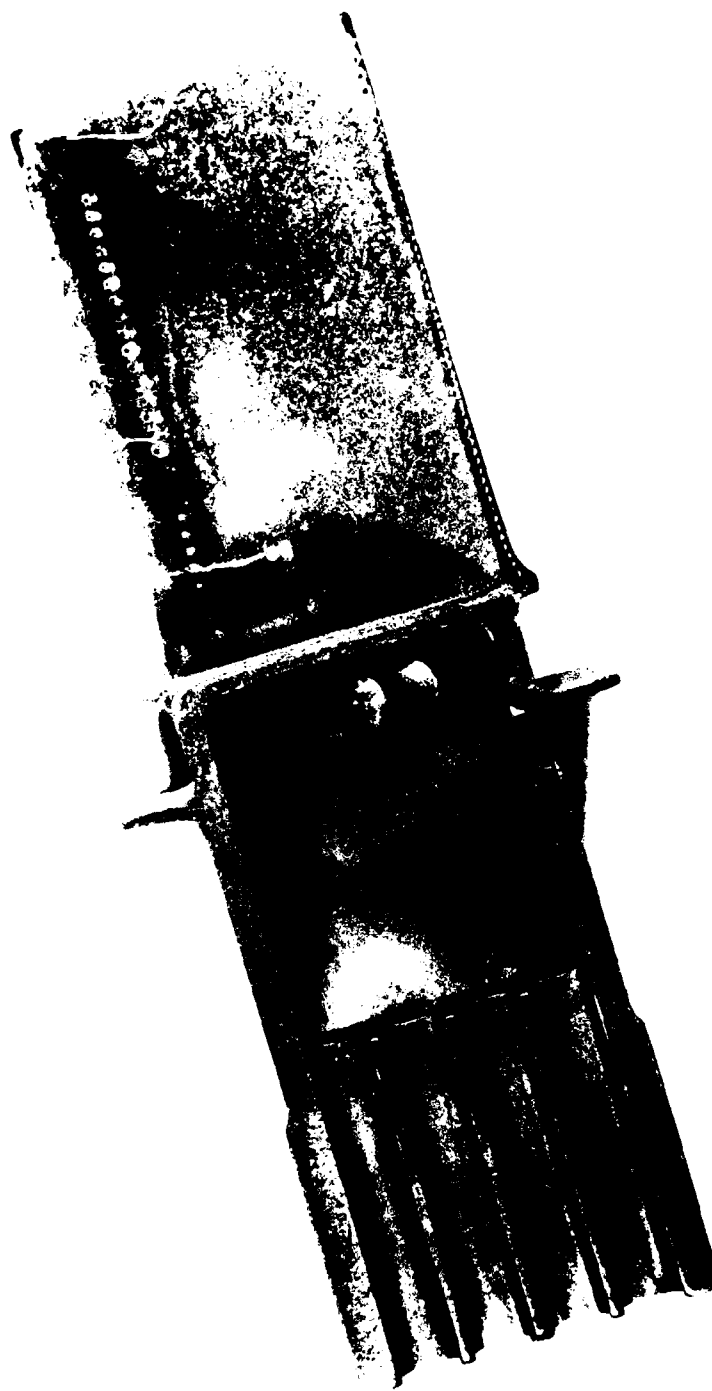Figure

Figure 28.   HP1 Compressor Blade
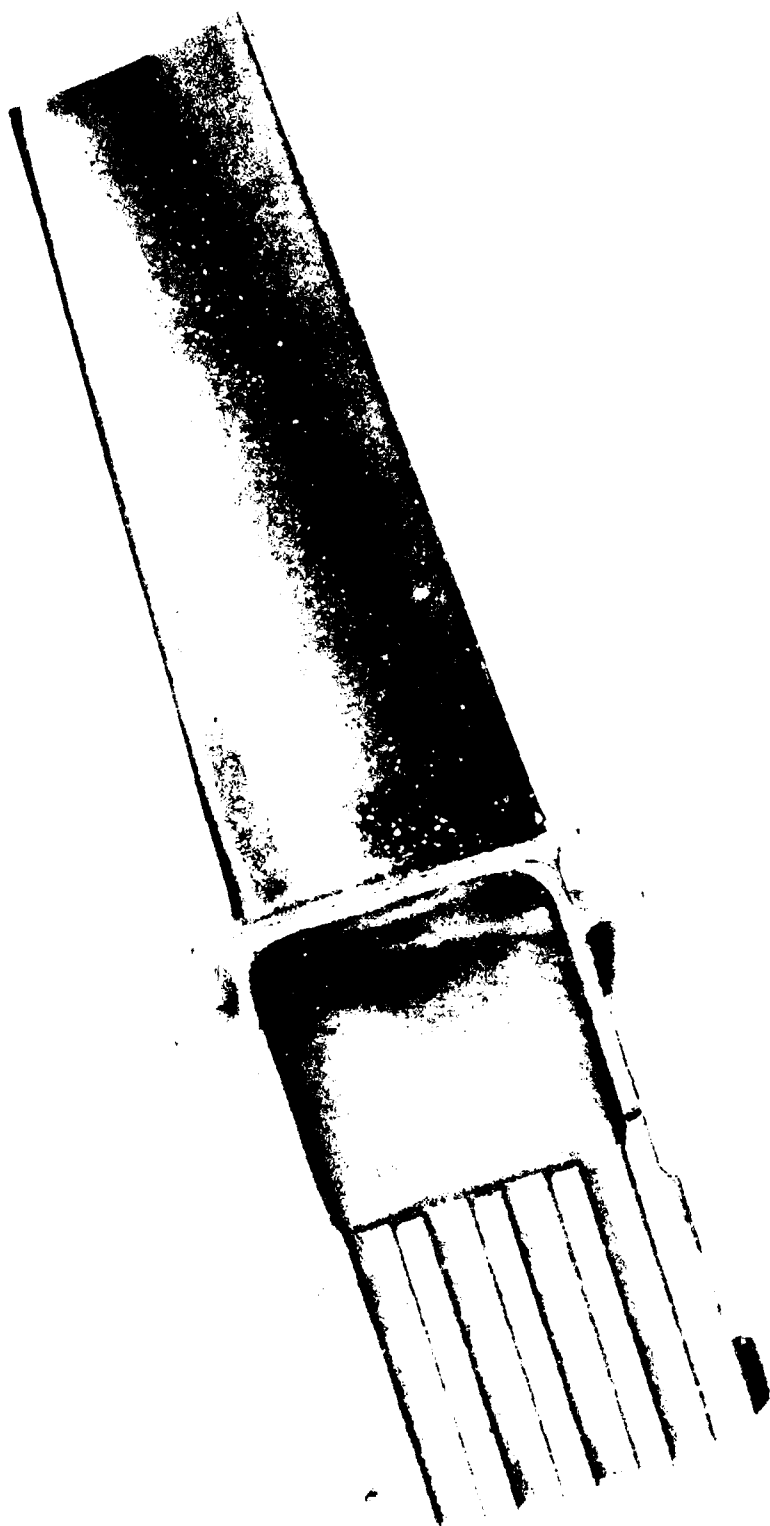
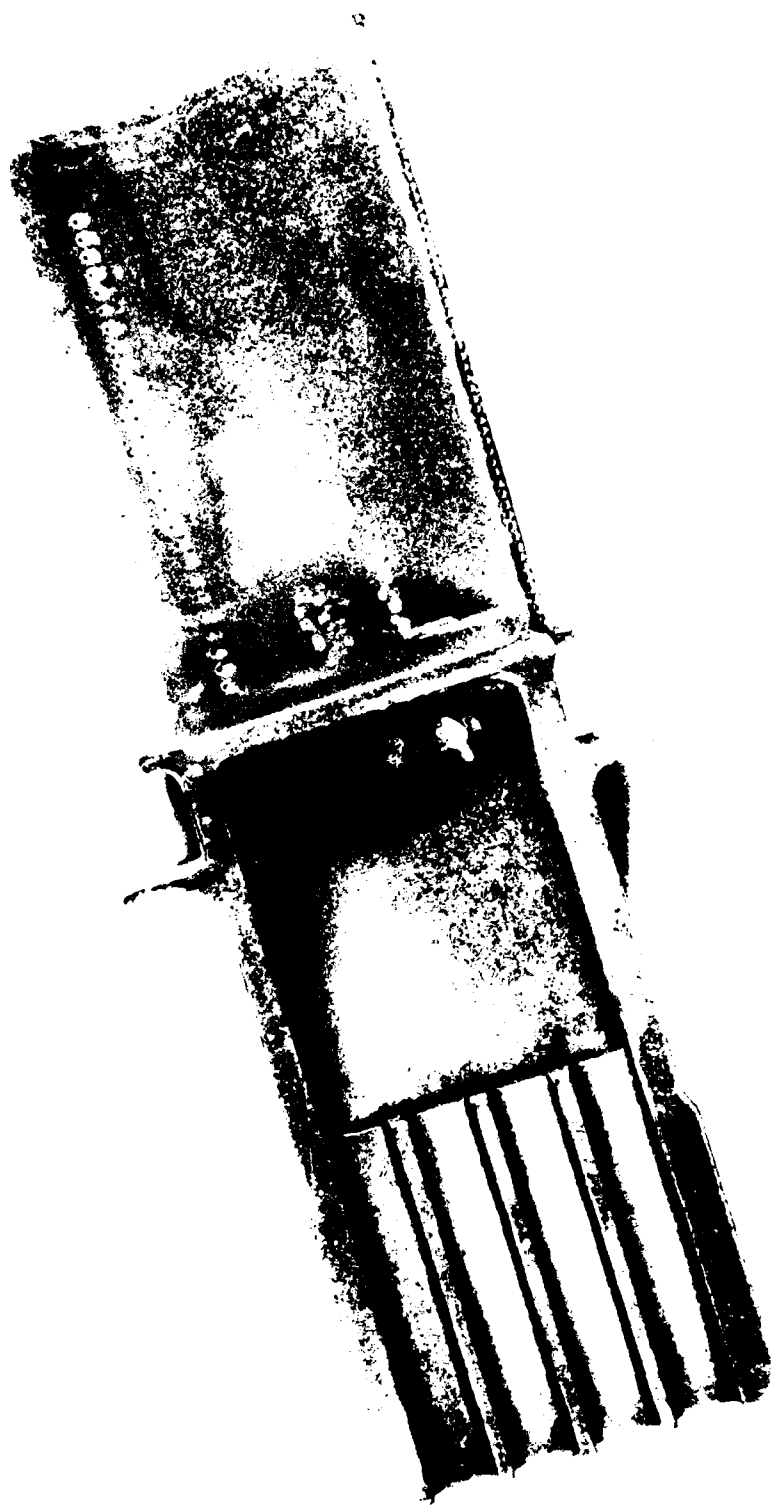Figure 32. 750° High Pressure Turbine Blade
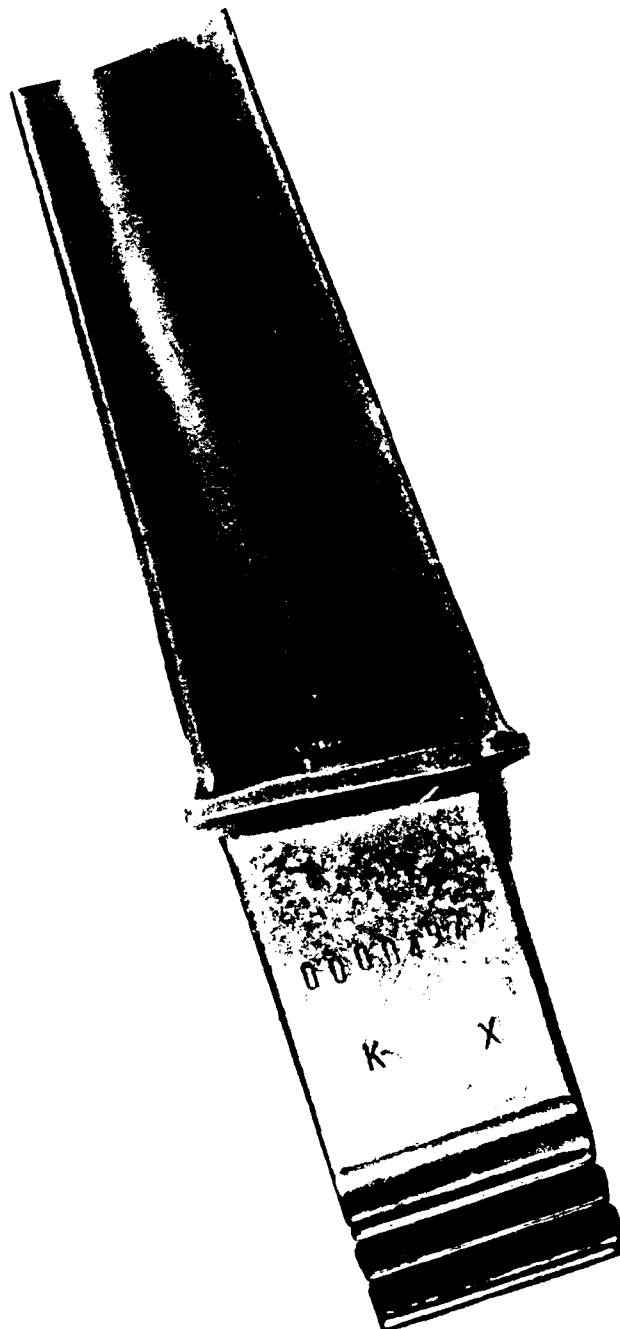
Figure 33. TF39 High Pressure Turbine Blade

Figure 34.   J79 Stage 1 Turbine Blade

Figure 15. F100 Compressor Blade

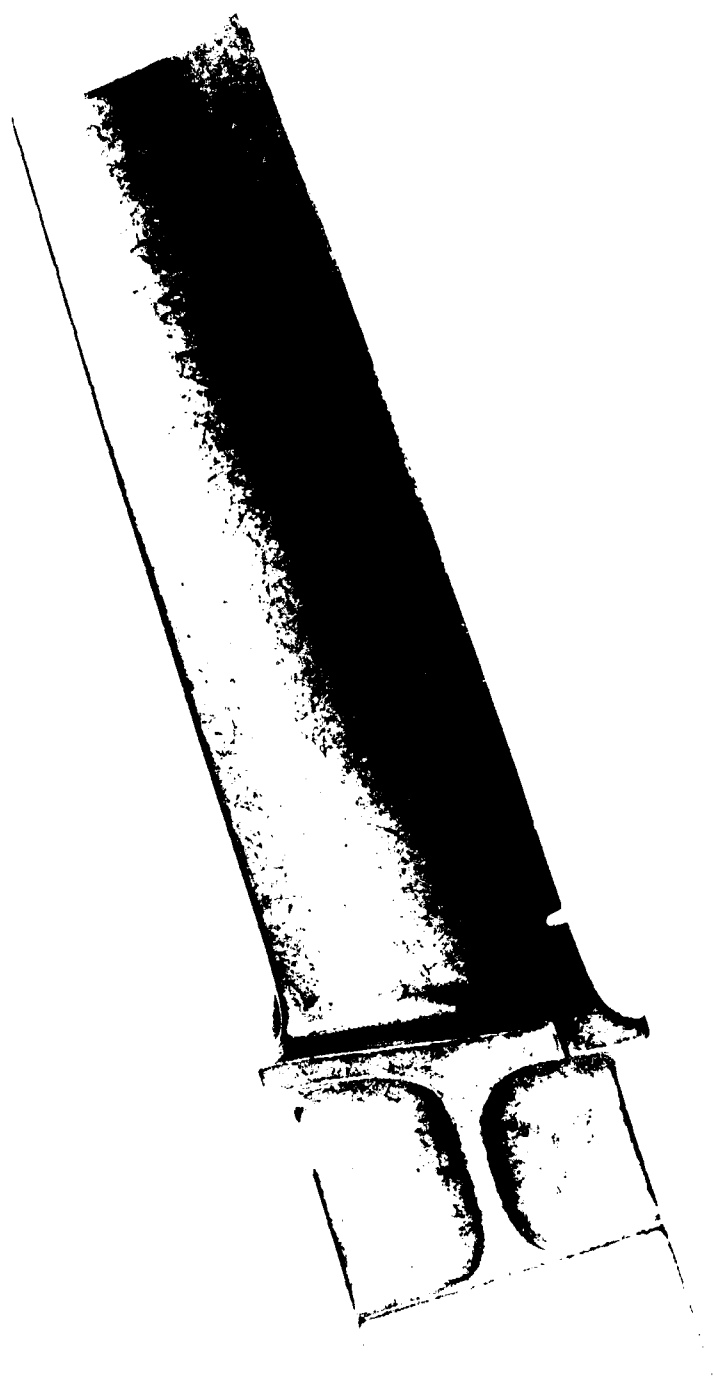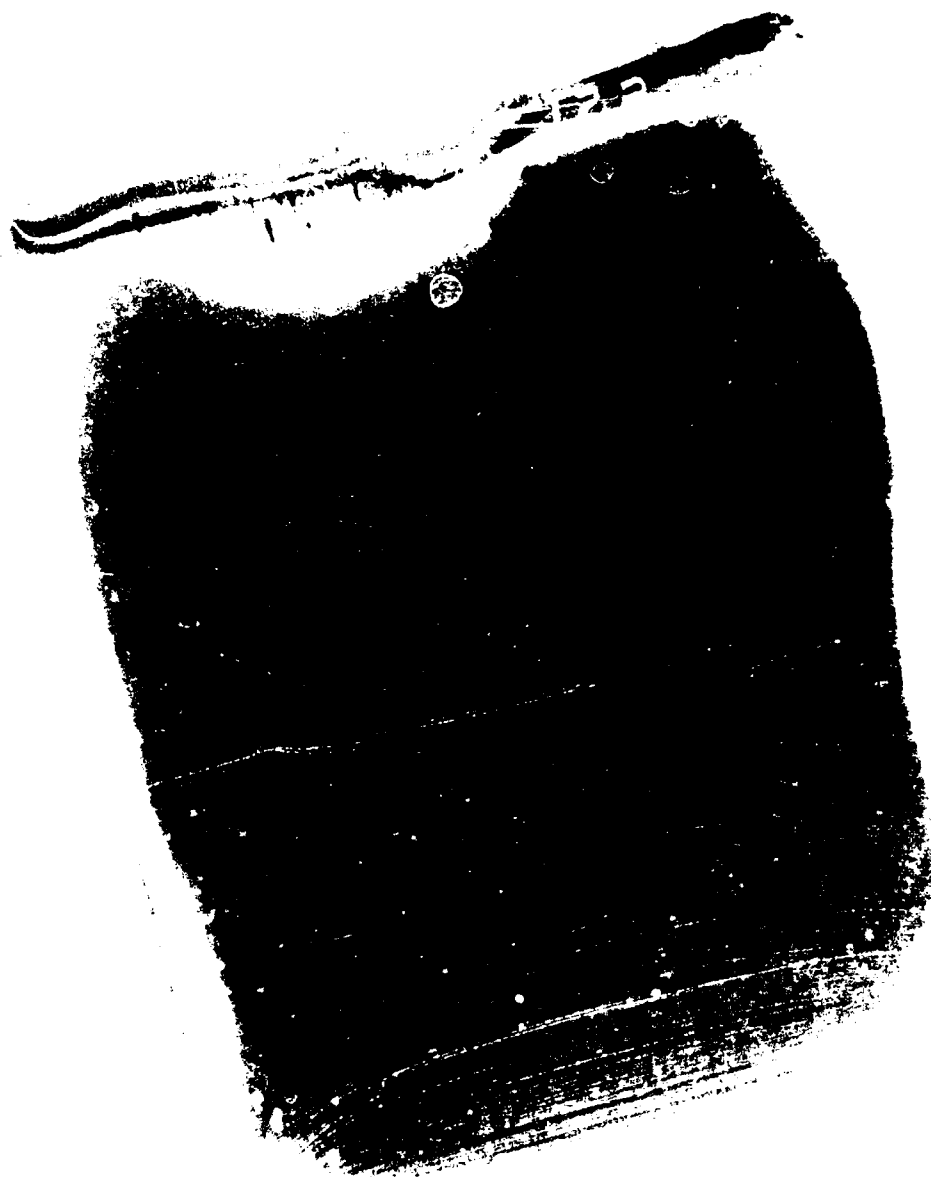Figure 36.   TF34 Stage 1 High Pressure Turbine Blade

Figure 47. J79 Stage 2 Turbine Blade

3.0 APPLICATION TECHNIQUES

3.1 EVALUATE APPLICATION TECHNIQUES

3.1.1 Select Statistical Experiment Design

In selecting the statistical experiment design for evalution of the various capsule and spary parameters consideration was given to optimizing the number of variables that could be effectively evaluated yet keep the amount of testing at a level where it could be accomplished within the program cost and time constraints. It was decided that a two level fractional factorial experiment design would best satisfy the needs of the program. A simple representation of the testing involved in a two-level fractional factored experiment is illustrated in Table 3. This experiment design allows a complete evaluation of the effect of eight factors and 13 two factor interactions. Interactions measured were between capsule diameter and all other factors and between spray pressure and the other seven factors. The resolution number of four listed in Table 3 refers to this fact that all of the primary factors and part of the interactions can be measured. A resolution of five is the highest with all the primary factors and all the resolutions being measured. The numbers -1 and 1 in the table ident fy the value of each factor being used for a particular test. For example, in identifying the diameter of the capsiles those having a 2-5μ diameter were designated -1 and those with a 20-40μ diameter were designated as 1. Due to the fact that 10 variables were identified as unwarranting evaluation the fractional factorial experiment was conducted three times. Thus each of the 15 airfoil tests specimens was inspected a total of 96 times.

3.1.2 Select Variables

Selection of variables for evaluation was done in conjunction with selection of the statistical experiment design. As the number of factors believed to have an effect on the encapsulated penetrant application process increased it becaue evident that a fractional factorial experiment design was required in orde to keep the effort within the time scope of the program. Even with a fractional factorial experiment design not a l factors identified were evaluated, to do so would have required a much lonper period of time than was originally scheduled. However, attempts were made to gain information on the effect of these factors, such as spray nozzle size and material feed cup pressure, on the sensitivity of the process. The factors selected for evaluation will be discussed in the following paragraphs.

In Section 2.2 of this report in discussing the preliminary production of encapsulated fluorescent penetrants, the three capsue parameters that were believed to play an important role in the sensitivity of the encapsulated penetrants were discussed. These factors were penetant, size of capsules, and capsule wall thickness. Since a two-level fractonal factorial experiment

Table 3. Fractional Factorial Experiment Design.

FRACTIONAL FACTORIAL EXPERIMENT DESIGN – TWO LEVELS

Number of Factors = 3   Resolution = 4
Number of Tests = 32

EXPERIMENTAL PATTERN FACTOR SETTINGS

Test #	A	B	C	D	E	F	G	H
1	-1	-1	-1	-1	-1	-1	-1	-1
2	1	-1	-1	-1	-1	1	-1	1
3	-1	1	-1	-1	-1	1	-1	-1
4	1	1	-1	-1	-1	-1	1	1
5	-1	-1	1	-1	-1	1	1	1
6	1	-1	1	-1	-1	-1	-1	-1
7	-1	1	1	-1	-1	-1	-1	1
8	1	1	1	-1	-	1	1	-1
9	-1	-1	-1	1	-1	-1	-1	1
10	1	-1	-1	1	-1	1	1	-1
11	-1	1	-1	1	-1	1	1	1
12	1	1	-1	1	-1	-1	-1	-1
13	-1	-1	1	1	-1	1	-1	-1
14	1	-1	1	1	-1	-1	1	1
15	-1	1	1	1	-1	-1	1	-1
16	1	1	1	1	-1	1	-1	1
17	-1	-1	-1	-1	1	-1	-1	-1
18	1	-1	-1	-1	1	1	1	1
19	-1	1	-1	-1	1	1	1	-1
20	1	1	-1	-1	1	-1	-1	1
21	-1	-1	1	-1	1	1	-1	1
22	1	-1	1	-1	1	-1	1	-1
23	-1	1	1	-1	1	-1	1	1
24	1	1	1	-1	1	1	-1	-1
25	-1	-1	-1	1	1	-1	1	1
26	1	-1	-1	1	1	1	-1	-1
27	-1	1	-1	1	1	1	-1	1
28	1	1	-1	1	1	-1	1	-1
29	-1	-1	1	1	1	1	1	-1
30	1	-1	1	1	1	-1	-1	1
31	-1	1	1	1	1	-1	-1	-1
32	1	1	1	1	1	1	1	1

design was selected this imposed a limit of only two values for each factor. As discussed in detail in Section 2.2 the penetrants encapsulated were ZL30A and RC-77 with a capsule diameter of 2.5 microns and 20-40 microns and both a thick and thin wall.

From preliminary work done at the Air Force Materials Laboratory it was known that spray pressure and the distance from spray gun to part had a significant effect on the sensitivity of the process. To select the two values of these parameters which were to be used in the fractional factorial experiment, preliminary investigation was performed. This involved spraying encapsulated penetrants on selected airfoil test specimens varying the pressure and distance. Although this was not a statistically designed experiment it became obvious that when the distance from the part to the spray gun was greater than four to six inches, even at high spray pressures (80 psi), the sensitivity was reduced. This, however, was also dependent on the size of the capsules. For the small diameter capsules the sensitivity was reduced with a distance greater than one to two inches. Therefore, two sets of values were selected that would allow us to effectively evaluate both the small and large diameter capsules; these were 1/2 and 1 inch, and 2 and 4 inches. This meant that the fractional factorial experiment had to be conducted twice.

Preliminary investigation also revealed that even at a small spray gun to part distance the sensitivity of encapsulated penetrants decreased with spray pressures below 40-50 psi. Thus, for most testing spray pressures of 60 and 80 psi were used except, as will be discussed later, when using the DeVilbiss EHP-603 powder spray equipment when pressures of 30 and 50 psi were used.

Other variables selected for evaluation included spray pattern, spray time, and electrostatic versus conventional spray. A small spray pattern or area was achieved in the conventional spray gun by closing the spreader valve such that the spray emitted by the gun was not effected, in the powder spray system this was achieved by setting the low vortex pressure at 0 psi. A large spray pattern or area was similarly achieved by setting the spreader valve at approximately three-quarters and in the powder spray system by setting the high vortex pressure at 30 psi.

Spray time is defined as the length of time a particular section of the part is being sprayed. When this time is complete the gun is moved to another section of the part and sprayed for a particular length of time. It does not refer to the total time required to completely spray a part. Spray times selected were one and two seconds.

3.1.3 Select Equipment

As with selecting candidate materials for encapsulation, the first task in selecting candidate spraying equipment was to contact the various manufacturers of spray equipment, then identify the material to be sprayed and receive their recommendations as to what would best satisfy the requirements. The following manufacturers were contacted: Binks, DeVilbiss, Graco, Parsche, and Aro. Of these the only one who responded positively was DeVilbiss. They suggested the EHP-603 Electrostatic Hand Powder Spray System. Photographs of the equipment are given in Figures 40 and 41. The system consists of a power supply which supplies electrical current to the electrostatic spray gun. The output current is negative polarity direct current and is ajustable from 0 to 90 kilovolts. The pneumatic control cabinet controls most functions of

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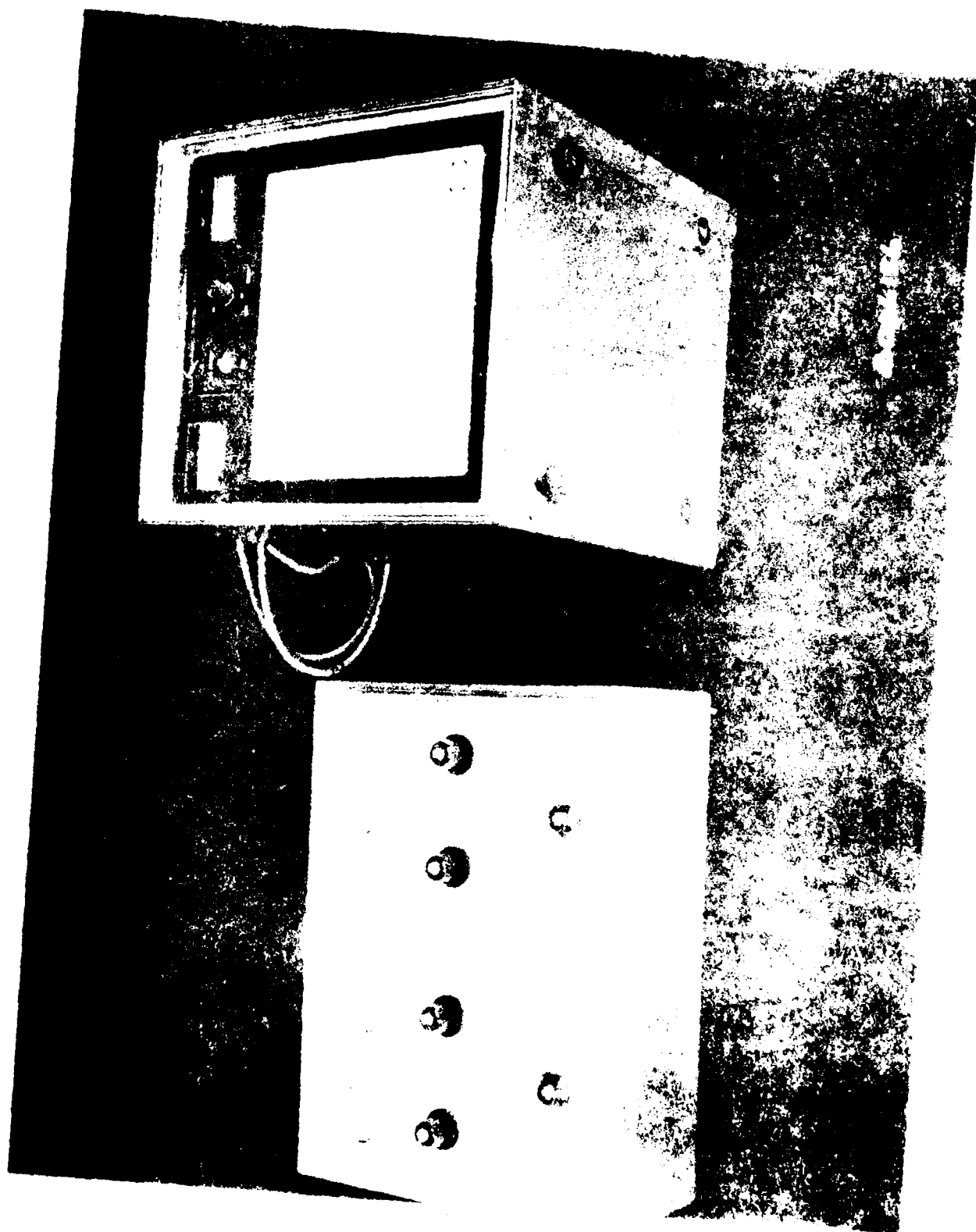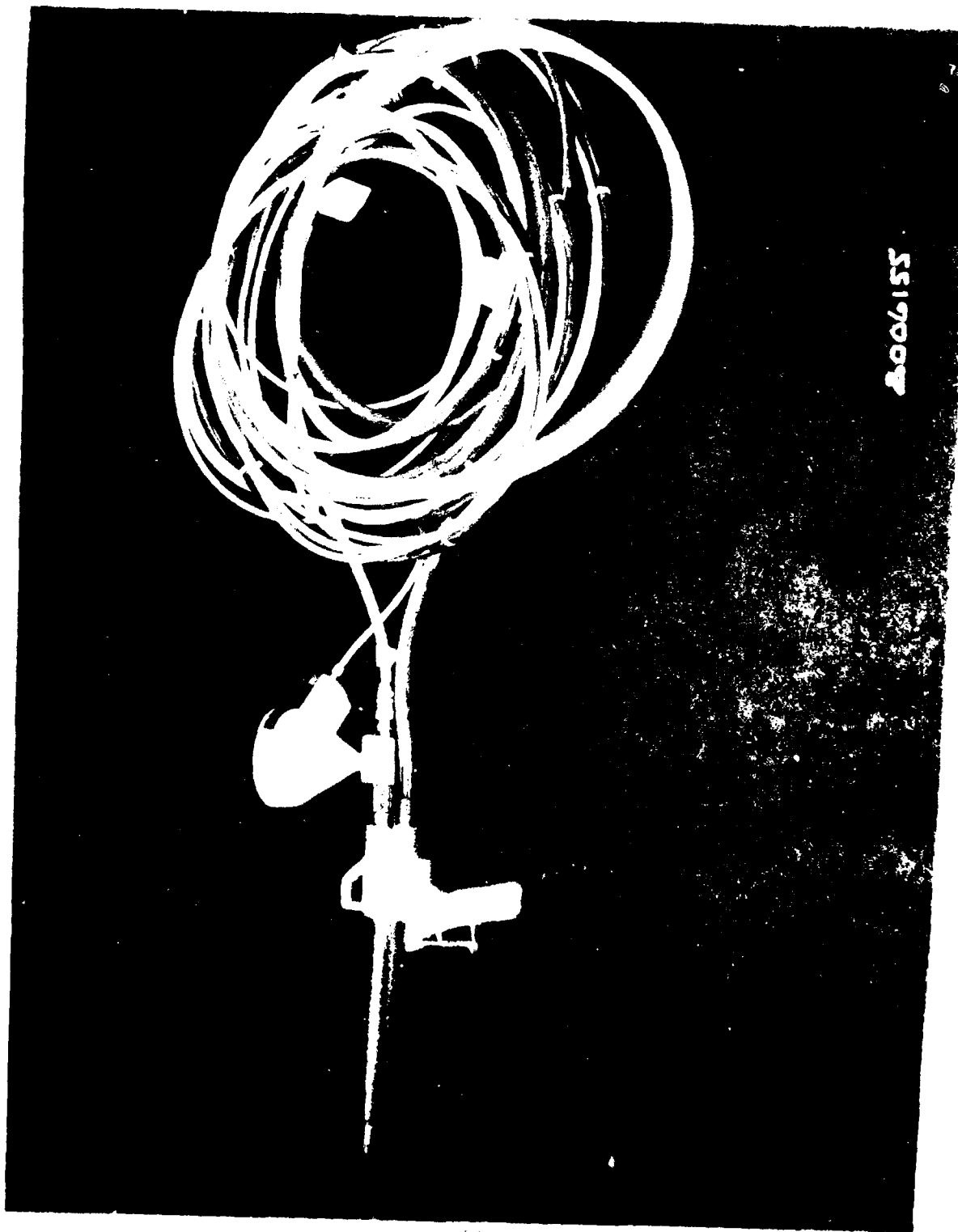
the electrostatic hand powder system. It provides air to the power supply to turn on and off current to the powder handgun, and air to the powder handgun to spray the powder. It includes pressure gages, controls for powder flow, and controls for supplying air to system components. The electrostatic powder handgun applies powder to the parts. It receives electrical current from the power supply and transfers it to the powder, causing the powder to be attracted to the article being sprayed. The micro feed powder supply is used when spraying small quantities of powders. A miniature combination venturi air pump and vibrator supplies a constant, uniform flow of powder from the supply cup to the gun. An air control knob on the cup regulates delivery rate of powder to gun. The electrostatic air cable interconnects the control cabinet, powder supply, and powder handgun.

Through initial evaluation and discussions with DeVilbiss it was determined that the powder spray system was limited by design to a maximum pressure of 50 psi. Therefore, we could not evaluate this system at the desired 60 and 80 psi spray pressure, thus, pressures of 30 and 50 psi with a spray distance of 1/2 and 1 inch were used.

DeVilbiss also provided a MBC spray gun with a pressure feed cup and a MGB spray gun with a suction feed cup. During initial evaluation it became evident that the suction feed was inadequate to provide a continuous flow of powder and thus it was eliminated from consideration. All evaluation of the MBC spray gun and pressure feed cup was done using a MBC-444-FX fluid needle and an AV-15-FX tip which has an opening of 0.042 inch. Towards the conclusion of the program a MBC-496-C fluid needle and AV-641-AC tip which has an opening of 0.110 inch was evaluated. There seems to be no significant change in the sensitivity of encapsulated penetrants due to the different tips; however, the frequent clogging that was experienced with the small tip size is significantly reduced when using the larger size.

3.1.4 Statistical Results

Having identified the capsule and spray parameters to be evaluated, the spray equipment to be used and the statistical experiment design, the next task was to conduct the experiment and evaluate results. As mentioned before the two level fractional factorial experiment was conducted three separate times. First, for the EHP-603 powder spray system, secondly, for the MBC spray gun and pressure feed cup using a spray distance of 2 and 4 inches, lastly, the MBC spray gun and pressure feed cup with a spray distance of 1/2 and 1 inch was evaluated. To compare results between each system additional spraying was done using the preferred parameters of each system as identified by the fractional factorial experiment and statistically comparing the three systems.

EHP-603 Powder Spray System - Results of the two-level fractional experiment are given in terms of percentages representing the contribution that each factor had on the total sensitivity of the process. Percentages less than three are not considered statistically significant. For the EHP-603 system

the main factors were: 20-40 micron capsule diameter constituting 87.8% of the total sensitivity, thin wall contributed 3.85%, encapsulated ZL30A penetrant had a percentage of 3.15%, and 50 psi pressure was slightly better than 30 psi with 3.00%. All other factors were not statistically significant; however, it would be most advantageous from an applications standpoint to use a large spray pattern and minimum spray time. This facilitates subsequent excess penetrant removal by minimizing the amount of penetrant on the surface of the part.

Pressure Feed System at 2 - 4 Inches - The significant factors identified for this system were: 20-40 micron capsule diameter was the main contributor with 85.8%, and a direct spray, as opposed to spraying at a 45° angle, contributing 8.3%. None of the remaining factors proved to be statistically significant. As with the EHP-603 system it would be most advantageous to maximize the spray area and minimize the spray time.

Pressure Feed System at 1/2 - 1 Inch - As with the previous two systems the one most important factor in determining the sensitivity of the process was the capsule diameter with the 20-40 micron capsules contributing 44.9% of the total sensitivity. Although the large diameter capsules are still significantly better than those having a small diameter the difference is not as great as it was with the greater spray distance. The only other significant factor in this system was that a spray distance of one-half inch contributing 3.3% was better than one inch. This may have been caused by an increased sensitivity of the small diameter capsules at one-half inch.

Comparison of the Three Systems - Using the preferred parameters as identified above for each system, two separate tests were conducted on the systems and statistically compared. Results of these tests are given in Table 4. The ENP-603 system missed a total of four defects and when compared with the MBC system at two inches the total length of indications was reduced by 6%. Both of these factors are significant and thus led to the conclusions that the sensitivity of the EHP-603 system is less than for the MBC system. Both MBC systems found the same number of defects and to total lengths of indications were comparable. At one half inch the length of indications was approximately 1% less than at two inches. This, however, is not significant and thus no statistical difference can be seen between these two systems.

3.2 RECOVERY AND REUSE

One of the most attractive potential benefits of an encapsulated penetrant system was the possibility of recovering and reusing almost 100% of the overspray. Effort to identify a method of recovering and reusing encapsulated penetrants concentrated on two fronts. A simple laboratory type spray enclosure was constructed that would allow investigation of the properties of the post-sprayed encapsulated penetrants and also allow recovery for reuse in this program. A photograph of a typical spray application using this enclosure is shown in Figure 42. The second effort was an industry search of potential and/or available means of recovering and reprocessing powders in the size range of interest.

Table 4. Sensitivity Comparison of Systems.

PART S/N	EHP-603 SYSTEM		MBC SYSTEM @ 2 INCHES		MBC SYSTEM @ 1/2 INCH	
	Run #1	Run #2	Run #1	Run #2	Run #1	Run #2
4	.030	.030	.030	.030	.030	.030
5	.020	.020	.020	.020	.020	.020
1	.250	.250	.300	.280	.280	.280
3	.020	.020	.020	.020	.020	.020
	.045	.045	.045	.045	.045	.045
12	1.000	1.000	1.000	1.000	1.000	1.000
	.650	.650	.700	.700	.650	.650
10	.250	.250	.300	.300	.300	.280
14	.400	.400	.400	.400	.400	.400
	.400	.400	.400	.400	.400	.400
15	.200	.200	.200	.200	.200	.200
	-	-	.060	.045	.045	.045
	-	-	.045	.030	.030	.030
	.250	.225	.260	.260	.250	.250
	.440	.400	.450	.460	.460	.440
	.300	.300	.300	.300	.300	.300
	.200	.200	.200	.200	.200	.200
16	.045	.045	.045	.045	.045	.045
	.020	.020	.030	.030	.020	.020
	.020	.020	.020	.020	.030	.030
	.010	.010	.010	.010	.010	.010
	.030	.030	.030	.030	.045	.045
17	.020	.020	.030	.030	.030	.030
	.045	.045	.045	.045	.045	.045
	.015	.020	.020	.020	.020	.020
	.020	.020	.030	.030	.030	.030
	.200	.200	.220	.220	.220	.200
7	.800	.800	.800	.800	.800	.780
18	.250	.250	.250	.250	.250	.250

3.2.1 Laboratory Findings

It became apparent early in the program that encapsula ed penetrants would have to be treated before they could be resprayed. Post-sprayed capsules tended to agglomerate significantly reducing their flowability and making respraying difficult. Part of this is caused by electrostatic attraction between capsules. This is easily alleviated by the addition of an anti-static agent to the sprayed capsules. Furthermore, at initial manufacture the capsules can be anti-statically treated which would minimize this attraction between capsules. Investigation also revealed that approximately 1% or
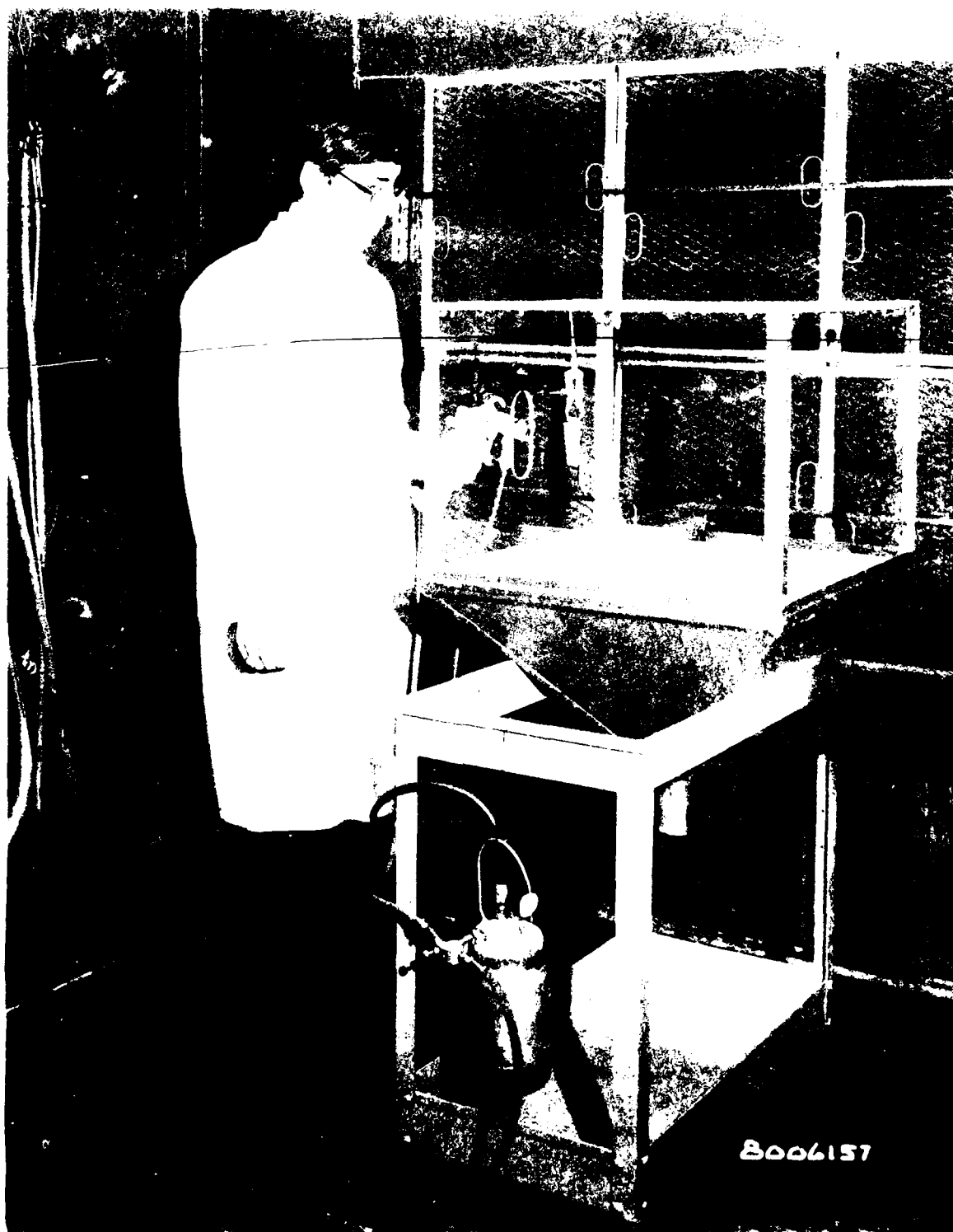
Figure 42.   Typical Laboratory Spray Application

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less of the capsules were breaking during the spraying process. This released oil mixed with the dry capsules causing the formation of aggregates. Before reuse this free oil needed to be removed from the system.

3.2.2 Capsulate Treatment Method

Samples of the post-sprayed capsules were submitted to the encapsulating manufacturers for evaluation of reconditioning methods. The following methods were identified. The first method involved simply adding a small amount of silica to the capsules. Silica absorbs the loose oil and the resulting capsules display very good flow characteristics. Before this method could be used certain aspects would have to be evaluated. The minimum amount of silica that should be added to make the flowability of the capsules acceptable, the effect the additional silica has on the sensitivity of the process, and the number of times capsules can be treated in this manner are questions which were beyond the scope of this program that should be addressed in subsequent effort.

The second method, although more involved than the one just described, may have the advantage of not reducing the life of the penetrants to the degree of the first method. Again, this is an aspect that needs further investigation. This method involves (1) washing the capsules with Freon TF to remove the free oil caused by ruptured capsules, (2) drying the capsules, (3) blending in a small amount of fused silica and an antistatic agent, and (4) dry sieving the capsules.

3.2.3 Production Type Recovery System

Industry search of powder recovery methods revealed that there are presently available complete closed loop systems for recovering, reconditioning, and respraying powders. Illustrated in Figure 43 is a schematic of a typical system offered by the DeVilbiss Company. The powder is applied in a spray booth with the overspray powder being exhausted to a collector, the powder then goes through a sieve and separator which rejects any large or foreign particles. New and used powder is introduced to the powder conditioning tank which in existing systems removes any moisture from the powder and makes it fluffy. For encapsulated penetrants, this powder conditioning subsystem might have to include a washing, drying, and sieving operation. From the powder conditioning tank, the powder is transferred to a powder feed tank and resprayed. An estimate of the recovery capability of such a system is between 95-98% of the overspray.

3.3 PILOT LOT PRODUCTION

As a result of the application technique evaluation described in section 3.1, acceptable capsule parameters were identified. With this information, a pilot lot production of encapsulated penetrant was ordered. The purpose of
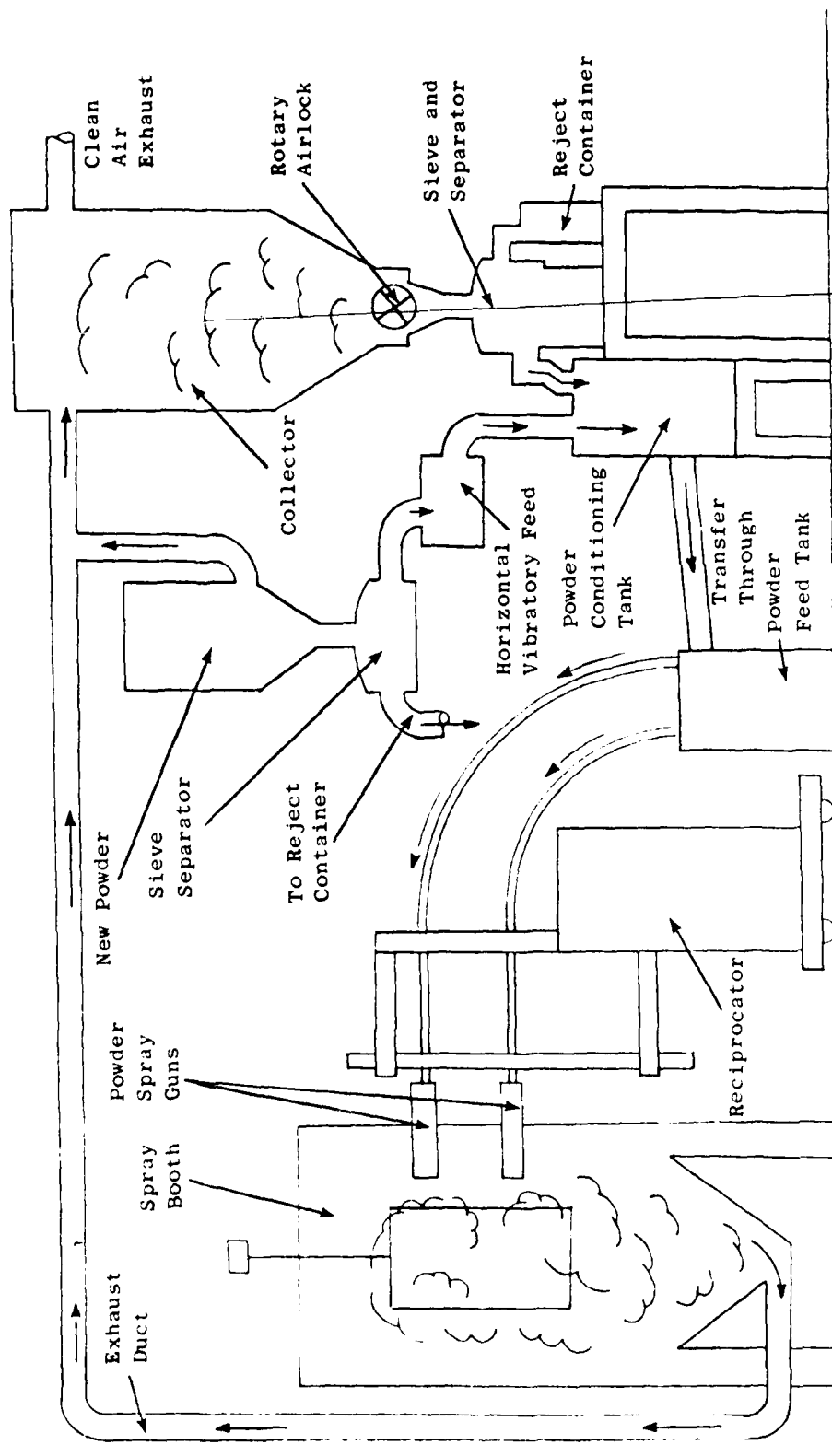
Figure 43. Typical Closed Loop Powder Conditioning, Supply and Recovery System.

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this pilot lot production was to demonstrate that the encapsulating process
could produce a consistent material and to provide sufficient material for
later production mode line testing.

The pilot lot material specified consisted of ZL30A in 20-40 micron
capsule diameter with a thick wall. Capsule wall thickness of this material
was slightly greater than for previous material, this also made the capsules
more rigid and thus more prone to rupture. Capsule breakage was increased
with this formulation. Originally it was thought that a thicker wall would
produce a stronger capsule; however, it now appears that the best capsule
configuration may be a compromise between the thickness and flexibility of
the wall.

4.0 REMOVAL AND DEVELOPER TECHNIQUES

4.1 EVALUATE REMOVAL TECHNIQUES

Although the encapsulated penetrant system uses a dry material, some particles do adhere to the surface of parts and create an objectionable background fluorescence. This excess material must be removed in order to produce an acceptable surface for inspection. It was originally anticipated that this excess material would be loosely held on the surface of the part and easily removed by blowing, brushing, or vacuum. However, it became evident after initiation of penetrant application that there was a strong adherence of the particles to the surface of the part. This is due to several factors. Some capsules will acquire a static charge as they exit the gun and thus be attracted to the part. At the high application pressure there is a compacting of capsules on the surface of the part. (At the lower pressures this effect was significantly reduced). A small percentage of the capsules will burst on impact with the surface of the part and release oil. This free oil must then be removed from the surface of the part.

Various methods were evaluated to determine the most effective removal procedure. Brushing with either a soft or stiff brush or wiping with a dry cloth removed most of the particles that were intact, but left a heavy fluorescent background which was unacceptable. Blowing with high pressure air (psi) also failed to significantly reduce the fluorescent background. Spraying with water at approximately 50 psi although better than the previous two methods still left a significant amount of background which was unacceptable. Dipping or wiping with a solvent such as 1-1-1 trichloroethane was adequate to remove most of the background but left fluorescent streaks on the surface of the part, but more significantly reduced the sensitivity of the process by removing penetrant from some of the capsules in discontinuities. Wiping the part with a water moistened cloth, although somewhat difficult and requiring a significant amount of pressure, would adequately clean parts having medium to fine surface finishes; however, results on parts with rough surfaces were poor. Spraying with a 5% solution of ZR10A emulsifier and water at 50-60 psi did a very good job of adequately removing the excess penetrant. Brushing or wiping with a mild detergent solution equally effective in eliminating the background fluorescence. This was very simple to do and required very little pressure. Most detergents would seem to do an adequate job. Those evaluated included a 5% solution of ZR10A emulsifier and a 1% or less solution of both Gentle Fels and Ivory dish washing liquids.

Certain things can be done to facilitate excess penetrant removal. These include minimizing the spray time to reduce the number of capsules that are left on the surface, treating the capsules with an antistatic agent to help reduce electrostatic attraction, and designing capsules that would be less susceptible to rupture.

4.2 EVALUATE DEVELOPER TECHNIQUES

Developers are employed in the conventional penetrant process to draw penetrant from indications so that they will fluoresce more brightly while still retaining their basic shape and thus be more visible to the inspector. A developer is required to improve visibility of flaws because in the conventional process some of the penetrant is usually removed from the flaw opening during the emulsification and rinsing procedures. It was initially proposed that the encapsulated penetrants would be held at the surface of a flaw after application and would not be as easily removed during the excess penetrant removal process. This should reduce the need for the use of developer. However, it became apparent during sensitivity measurements that developing would be required to achieve MIL-I-25135 Group VI sensitivity. However, it was not expected that developers would behave in the same way on encapsulated penetrants as they do with liquid penetrants. For example, dry developers would be expected to display poor sensitivity since they rely on free oil to "wick" the penetrant from the discontinuities.

Several developing techniques were evaluated to determine which were the most effective in achieving the required sensitivity. Photographs of a fine and coarse-cracked chrome panel with D-100 developer on one half and other candidate developers on the other side are included in figures 44 through 49. D-100 developer was used as a standard throughout the program.

The highest sensitivity was achieved with the use of nonaqueous wet developers. These were used in aerosol cans which are readily available through the various vendors. As can be seen from the photographs the sensitivity of Sherwin's Dubl-Chek D-100, Turco's Fluro-Check NAD, Magnaflux's Spotcheck SKD-HWA, and Tresco's D4990 are equivalent. Subsequent sensitivity measurements showed that these sensitivities were equal to a MIL-I-25135 Group VI penetrant system. The only nonaqueous wet developer evaluated that did not display good sensitivity was Met-L-Chek D-70. Nonaqueous wet developers apparently work well on encapsulated penetrant because the solvent which holds the developers in suspension can either penetrate the capsule walls and "free" the penetrant from the capsules or the solvents may actually break the capsule wall and release the oil. The developer particles on the surface can then "wick" the oil from the discontinuities.

Magnaflux's ZP-4B dry developer, although it did improve the sensitivity of the encapsulated penetrants, was not as good as the nonaqueous wet developers and not equivalent to a MIL-I-25135 Group VI penetrant system. Dry developers have a limited effectiveness with encapsulated penetrants because some of the capsules will rupture as they hit the rough edges of a discontinuity and release the oil. It is this free liquid penetrant which is now at the discontinuities that the dry developer is bringing to the surface.

The use of solvents alone proved to be very poor developers. Little wet penetrant from the capsules was brought to the surface as anticipated. If the penetrant was on the surface of the part there was no increase in brightness as with nonaqueous wet developers. The penetrant therefore tended to remain on the surface of the part leaving very poor indications.
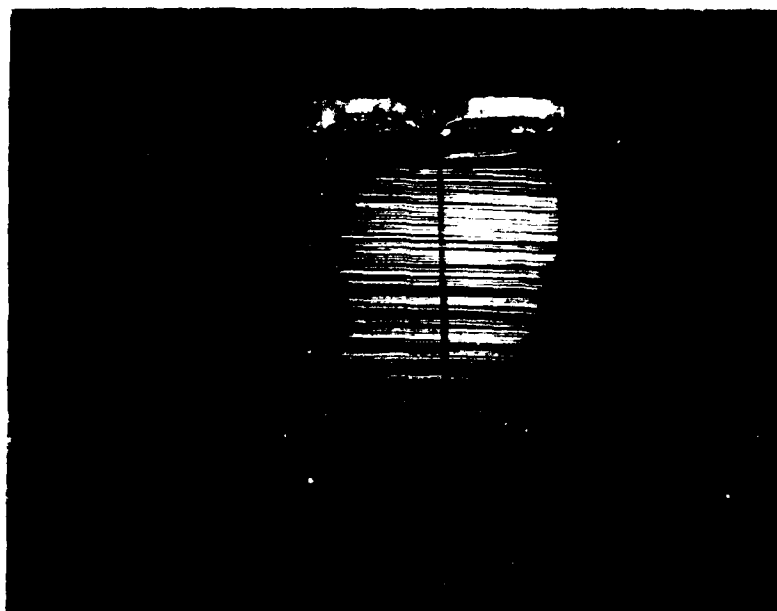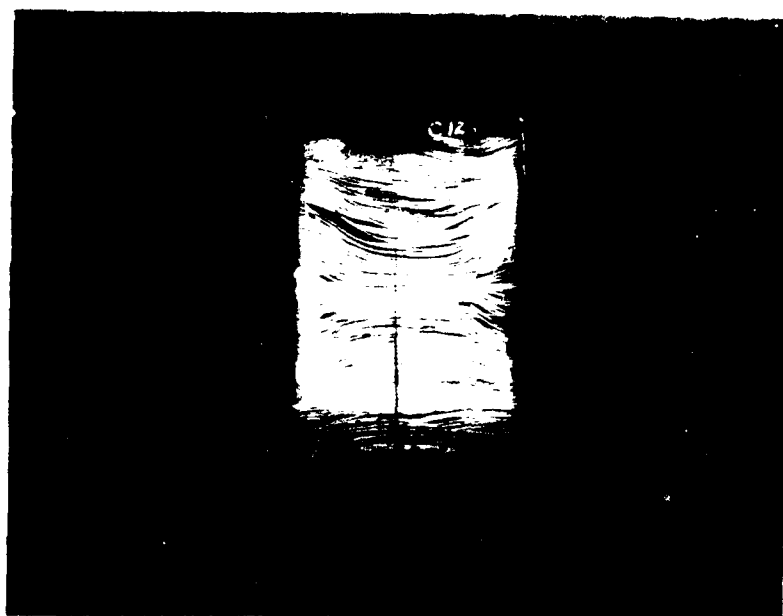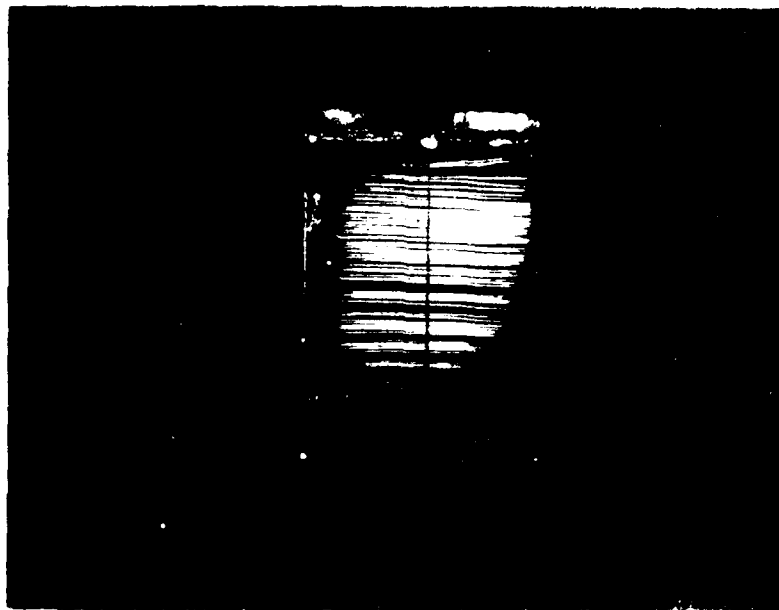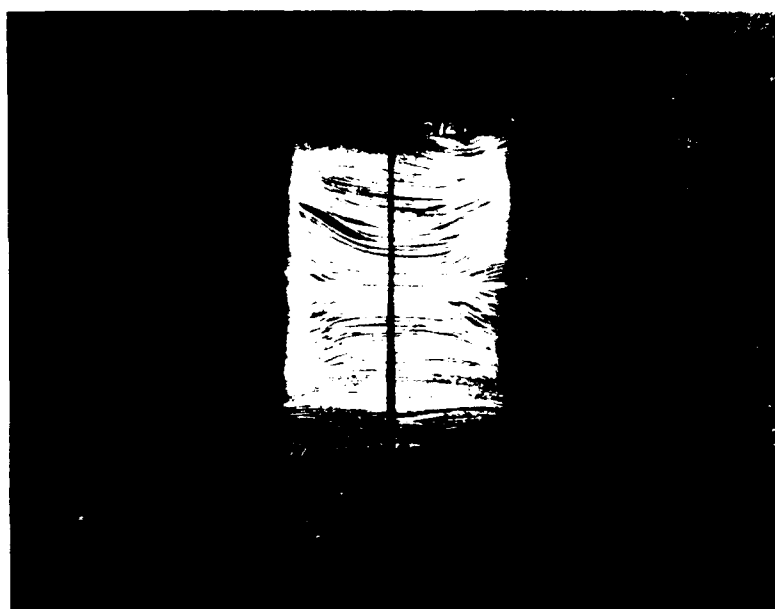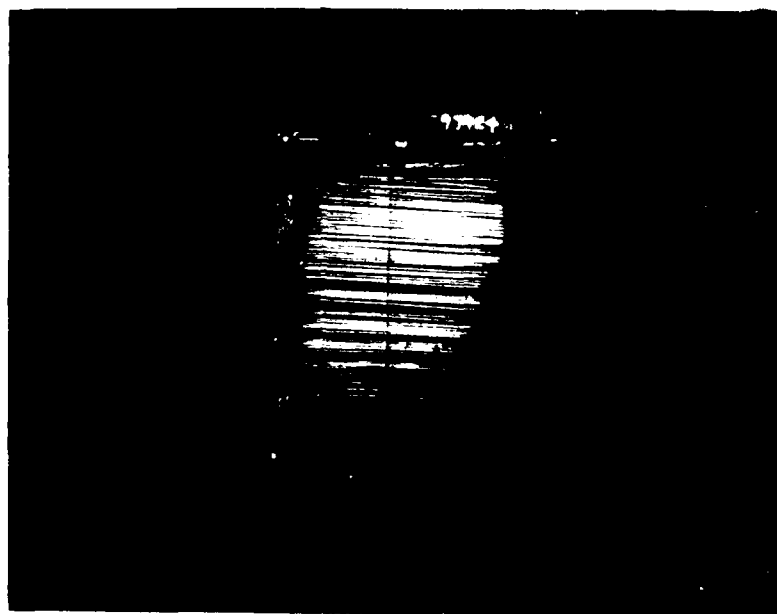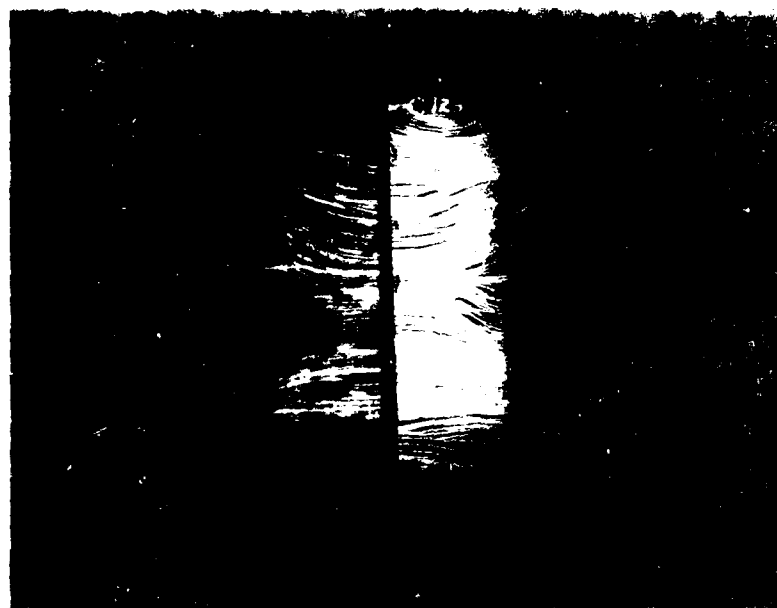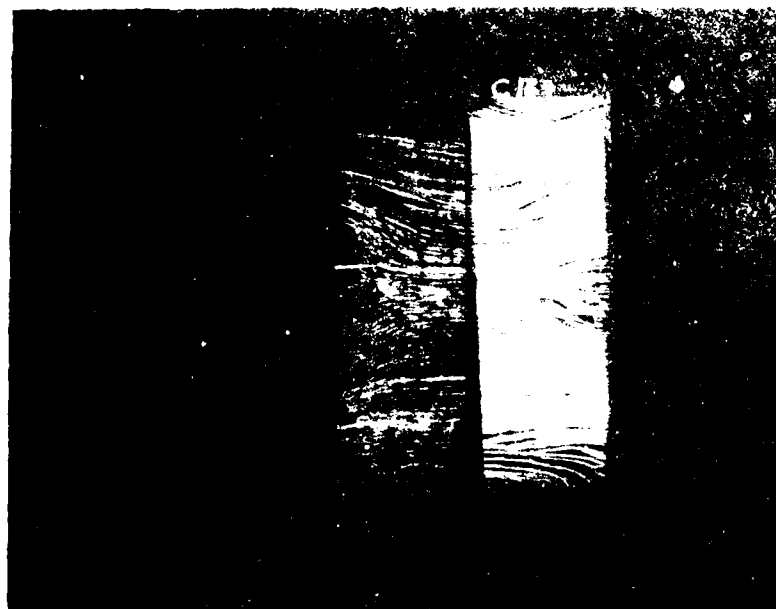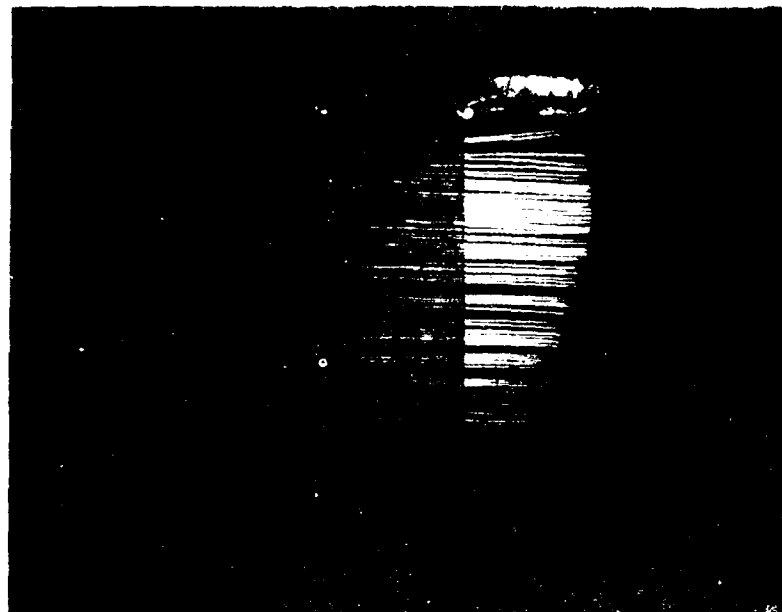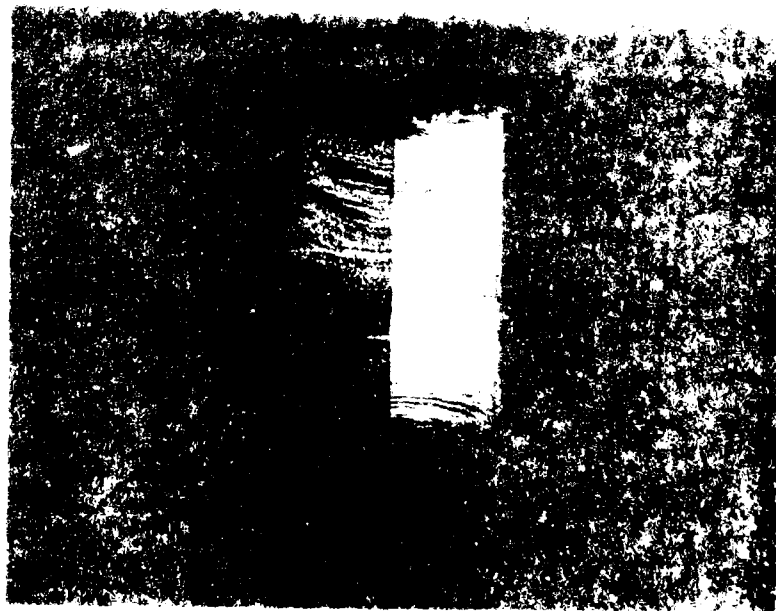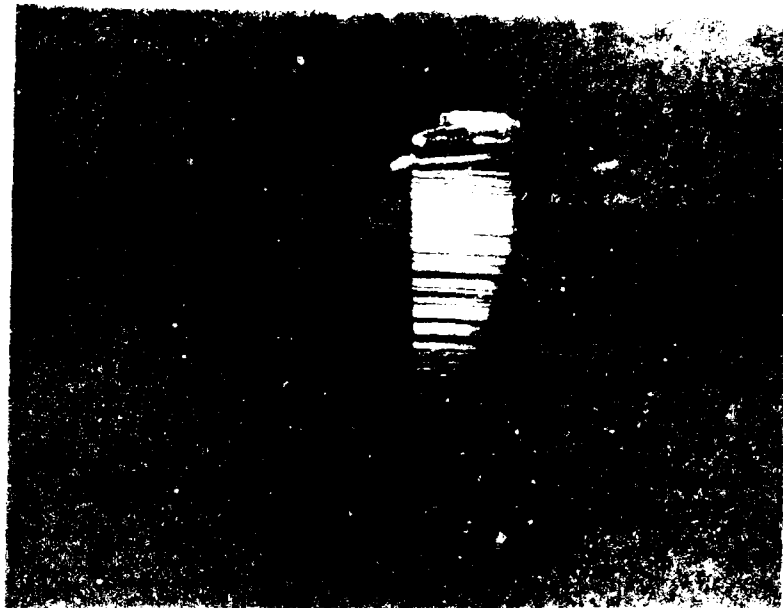
At present the developers that provide the required sensitivity are the nonaqueous wet developers. However, there should be potential for other developers that would be softened by other solvents, such as water. This would eliminate the need for nonaqueous wet developers. It is also conceivable that an electronic sensor might be used to scan the part for indications and no developer would be required because of an increased sensitivity over the human eye.

5.0 PROCESS DEMONSTRATION

5.1 SCOPE

The purpose of this phase of evaluation was to demonstrate a conventional MIL-I-25135 Group VI liquid penetrant system and an equivalent sensitivity encapsulated penetrant system in a simulated production line mode. From this demonstration part throughput and economics of each system would be estimated. Three systems were compared; an automatic liquid penetrant system such as has been proposed for the IBIS "Automatic Fluorescent Penetrant Processing Facility" (AFPPF); an automatic encapsulated penetrant system; and a manual liquid penetrant process such as is presently used at the Air Logistics Centers (ALC). Additionally in this phase of evaluation the environmental impact of using encapsulated penetrants was assessed.

5.2 MANUAL LIQUID PROCESS

Presently the fluorescent penetrant procedure used at the Air Logistics Centers involves using a liquid penetrant in conjunction with a lipophilic emulsifier. In this system the liquid penetrant is applied to a part or basket of parts by either dipping or spraying. After a 30 minutes dwell time in which the excess penetrant is allowed to drain off the part and back into the tank, the part is completely *submerged in the lipophilic* emulsifier removed and allowed to dwell for thirty to ninety seconds. The part is then rinsed and checked under a black light to assure that any excessive fluorescent background has been removed. Following this, the part is submerged in, or sprayed with, a water-soluble developer and placed in an oven to dry. When the part is completely dry it is ready for inspection in a darkened room with a black light.

This process requires a relatively small initial investment. However, there are disadvantages to this system. The lipophilic emulsifier mixes very easily with the penetrant and thus the emulsifier dwell time needs to be carefully monitored to assure proper sensitivity. Rinsing after emulsification also needs to be carefully controlled as over washing does reduce the sensitivity of the process. A lipophilic emulsifier is a relatively thick oil and a considerable amount is dragged out when the part is removed. Most of this is lost in the rinsing operation, thus making the emulsifier cost relatively high.

5.3 AUTOMATIC LIQUID PROCESS

The proposed "Automatic Fluorescent Penetrant Processing Facility" (AFPPF) is a fully automated, programmable system consisting of the following 16 stations. At a load station, individual parts will be placed in fixtures or groups of parts in baskets and subsequently loaded into the conveyor system. The fluorescent penetrant application station will electrostatically spray a

penetrant on the parts. Following penetrant aplication the parts will enter
the fluorescent penetrant dwell station where the penetrant is allowed to
dwell on the parts for a minimum of 30 minutes. This is followed by the pre-
rinse station where most of the excess penetrant is removed from the surface
of the parts. This being accomplished the parts then proceed to the hydro-
philic remover station where they are continuously sprayed or submerged in an
agitated solution for a maximum of 90 seconds. The solution can range from a
minimum of 3% concentration of remover to a maximum of 50% depending on system
requirements. After the appropriate emulsification time the parts are again
rinsed to remove the emulsifier. If the parts are to receive a water soluble
developer they then proceed to the wet developer station followed by the dryer
station. However, if dry developer is to be applied to the parts the wet de-
veloper station is bypassed and the parts go to the dryer station followed by
the dry developer station where the developer is electrostatically applied.
Finally, the parts are unloaded and are inspected.

A significant advantage of such a system is the greatly increased part
throughput. Further, human errors are eliminated thus increasing the overall
consistency of the process. Initial equipment cost including computer hard-
ware and software is relatively high for such a system.

5.4 AUTOMATED ENCAPSULATED PENETRANT PROCESS

Encapsulated penetrants would be most efficiently utilized in a com-
pletely automated system. In such a system, the parts would be individually
loaded on a fixture in an automated, programmable conveyor. Processing of
parts in groups, such as in baskets, is not believed to be feasible since all
surfaces of the part need to be accessible. Penetrant application is accom-
plished by spraying the capsule on to the part at high pressures (60-80 psi)
at a short distance (4 inches maximum). The parts and/or nozzle may need to
be manipulated such that all surfaces of the parts are exposed to a direct
spray. Part of the penetrant application station would be the powder re-
covery, reconditioning, and reuse system described in Section 3.2.3. Excess
penetrant removal may be accomplished by automatic brushing, wiping, or spray-
ing with a light detergent solution followed by a post-rinse to remove the
detergent. The parts would then be dried and a nonaqueous wet developer ap-
plied. A nonaqueous wet developer is presently necessary to attain the re-
quired MIL-I-25135 Group VI sensitivity. Parts are then unloaded and in-
spected.

As with the automated liquid process the main advantages of this system
are an increased part throughput and the reduction of human error. The en-
capsulated penetrant system also has the added advantage of eliminating the
30 minutes dwell time. Based on present knowledge, the main disadvantage of
such a system would be inability to process parts with complicated geometry,
the need to individually manipulate the parts, and the need to use nonaqueous
wet developers. The cost of such a system should be similar to an automated
conventional penetrant system.

5.5 ECONOMIC ANALYSIS

5.5.1 Assumptions

Economic evaluation of the above three systems was divided into three parts; material cost, labor cost, and equipment cost. A TF-39 Stage 6 compressor blade and TF-39 Stage 1 high pressure turbine blade were used to determine average spray time and usage rate. These blades were chosen since they represent different geometric complexities, the compressor blade being relatively simple, and an average size. For the automated systems the penetrant application time was assumed to have the longest lead time and thus became the limiting item determining part throughput. The required performance of the AFPPF was used in determining spray time and thus part throughput. It was assumed that with sufficient spray nozzles and part manipulation part throughput for the automated encapsulated penetrant system can be made the same as for the conventional liquid penetrant system. Part throughput for the manual liquid penetrant system is the average throughput being experienced at the Air Logistic Centers. Cost of liquid penetrant, emulsifier, and developer is the current price for bulk purchases of these materials. Assumed cost of encapsulated penetrants is the lowest cost currently quoted by the supplier for production quantities (1000 lb or more) of a MIL-I-25135 Group VI liquid penetrant. In the automatic liquid penetrant system both the penetrant and developer are assumed to be electrostatically applied.

5.5.2 Equipment Costs

Present estimates are that the initial cost of equipment for both the automated liquid and encapsulated penetrant systems will be equivalent. The savings that the encapsulated penetrant method realizes by the elimination of certain stations, such as penetrant dwell and prerinse, are offset by the higher cost of the penetrant application, recovery, and reuse station. It is assumed that the computer hardware and software would be equivalent for both systems. Approximate equipment cost for either system is in the range of $400,000 to $500,000. Since no computer or special conveyor is required for the manual liquid system, the initial equipment cost is significantly reduced. Present estimates of the cost of such a typical system is approximately $60,000.

5.5.3 Material Costs

5.5.3.1 Penetrant

5.5.3.1.1 Manual Liquid System

Penetrant is applied by dipping or by spraying but electrostatic application is not used.

    Average material usage - 0.0017 gal/part (measured)
    Cost of ZL30A - $20.24/gallon (55 gallon drum)
    Cost of penetrant - $0.034/part

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5.5.3.1.2 Automatic Liquid System

It is assumed that no penetrant will be recovered for subsequent reuse.

Flow rate - 0.027 gal/minute (manufacturer data)
Average spray time - 2.4 seconds
Number of spray nozzles - 2
Amount material required - 0.0022 gal/part
Cost of ZL30A - $20.24/gallon (55 gallon drum)
Cost of penetrant - $0.045/part

5.5.3.1.3 Automatic Encapsulated System

It is assumed that 96% of the capsules are recovered.

Flow rate - 0.07 oz/second (measured)
Average spray time - 2.4 seconds
Number of spray nozzles - 4
Amount material required - 0.672 oz/part
Material lost (assume 4%) - 0.0269 oz/part
Cost of encapsulated ZL30A - $18.66/lb.
Cost of encapsulated penetrants - $0.031/part

5.5.3.2 Remover

5.5.3.2.1 Manual Liquid System

Average usage rate - 0.0005 gallon/part (measured)
Cost of ZE4A Emulsifier - $10.31/gallon (55 gallon drum)
Cost of emulsifier - $0.0052/part

5.5.3.2.2 Automated Liquid System

Average usage rate - 0.0003 gallon/part (measured)
Cost of ZR10A Remover - $9.09/gallon (55 gallon drum)
Cost of 20% dilution - $1.82/gallon
Cost of remover - $0.00055/part

5.5.3.2.3 Automatic Encapsulated System

Average usage rate - 4.0 fl oz/part
Cost of detergent - $8.04/gallon (5 gallon drum)
Cost after 0.2% dilution - $0.016/gallon
Cost of detergent - $0.0005/part

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5.5.3.3 Developer

5.5.3.3.1 Manual Liquid System

Average usage rate - 0.0005 gallon/part (measured)
Cost of ZP13A water soluble developer - $1.15/lb (100 lb lots)
Dilute 1 lb powder with 1 gallon water
Cost of water soluble developer - $0.00058/part

5.5.3.3.2 Automatic Liquid System

Average usage rate - 0.1 gram/part (measured)
                - 0.00022 lb/part
Cost of ZP4B dry developer - $2.55/lb (25 lb bags)
Cost of dry developer - $0.00056/part

5.5.3.3.3 Automatic Encapsulated System

Flow rate - 8 fl oz/min. (manufacturer data)
Average spray time - 2 seconds (measured)
Number of spray nozzles - 2
Amount material required - 0.00416 gal/part
Cost of D499C nonaqueous wet developer - $20.00/gallon
Cost of developer - $0.0832/part

5.5.4 Labor Costs

In evaluating labor costs the hourly rate is that presently used at ALC's.

Part throughput - 1.15 parts/minute
Hourly rate - $11.64/hr
Labor cost - $0.168/part

5.5.4.1 Automatic Liquid System

Part throughput - 25 parts/minute
Hourly rate - $11.64 hr
Labor cost - $0.0078/part

5.5.4.2 Automatic Encapsulated System

Part throughput - 25 parts/minute
Hourly rate - $11.64/hr
Labor cost - $0.0078/part

5.5.5 Summary

The table below summarizes the labor and material per part cost for the three systems:

	Manual Liquid	Automated Liquid	Automated Encapsulated
Labor	$0.168	$0.0078	$0.0078
Penetrant	$0.034	$0.045	$0.031
Remover	$0.0052	$0.00055	$0.0005
Developer	$0.00058	$0.00056	$0.0832
Total	$0.2078	$0.0539	$0.1270

As can be seen the automatic liquid penetrant system would be the most cost effective followed by encapsulated penetrants and finally the manual liquid system. The cost of the encapsulated penetrants was based on encapsulated ZL30A; other dye solutions have been successfully encapsulated with the resulting cost being approximately 35% lower. Encapsulation manufacturers are also giving serious consideration to modifying the structure of the capsule wall which will allow the dye penetrant to be released with less costly materials than conventional nonaqueous wet developers.

5.6 ENVIRONMENTAL IMPACT

General Electric's Industrial Hygiene and Environmental Quality Organizations were asked to evaluate and identify the potential health and environmental problems of using encapsulated penetrants. Neither organization foresaw any problem in approving the use of encapsulated penetrants when used in conjunction with an exhaust and recovery system. Since the encapsulated penetrant would be reused, the exhaust and recovery of the powder would already be an integral part of an encapsulated penetrant system. Some degree of free spraying (without recovery) would be allowed, such as in using an aerosol can, but due to the relative ease of the capsules to become airborne it would be necessary to use an exhaust system in all instances. Encapsulated penetrants are no more or less toxic than liquid penetrants thus the same restrictions apply to both systems.

6.0   CONCLUSION AND RECOMMENDATIONS

It has been demonstrated that the sensitivity of encapsulated penetrants is equivalent to a MIL-I-25135 Group VI liquid penetrant system. Application parameters and techniques for achieving this sensitivity have been identified. Demonstrated advantages of encapsulated penetrants over the conventional liquid penetrants include a significant reduction of penetrant bleed out from cooling air holes in high pressure turbine blades. This bleed out problem makes liquid penetrant inspection of the area surrounding the holes difficult. Liquid penetrants must be completely processed and inspected within a specified period of time in order to achieve their full sensitivity. One of the advantages of encapsulated penetrants is that if developer is not applied immediately the parts can be stored for a period of days or weeks and then developed with no apparent loss in sensitivity. Another advantage of encapsulated penetrants is the elimination of the thirty minute penetrant dwell time. Since encapsulated penetrants do not rely on capillary action to enter the discontinuities but rather are forced at high pressures into the cracks the excess surface penetrant can be removed immediately after application.

Potential advantages of encapsulated penetrants have also been identified. Further evaluation would be required to demonstrate feasibility of these applications. Presently liquid penetrants can not be used on certain plastics because the oils attack them. Encapsulated penetrants applied as a dry powder would eliminate this problem. Further, if capsule breakage does occur, a fluorescent material that is not harmful to plastics can be encapsulated. Liquid penetrants have to be used within a certain temperature range. At low temperature the capillary action is reduced and the oils may not enter the discontinuities and at high temperatures some of the volatile solvents in the penetrants will evaporate thus reducing the fluorescence of the penetrant. Since encapsulated penetrants do not rely on capillary action to enter discontinuities, using them at low temperatures should pose no problem. The oil in the encapsulated penetrants is completely enclosed thus no solvents can evaporate at high temperatures and therefore the fluorescence of the penetrant is not changed.

Work performed to date indicates several disadvantages of encapsulated penetrants over the conventional liquid process. As stated before encapsulated penetrants rely on pressure to force the capsules inside the discontinuities. To attain the required sensitivity a relatively direct spray must be applied to all surfaces of the part, thus all surfaces of the part must have line of sight accessibility. This would make parts with complicated geometry very difficult or impossible to process. Even parts with simple geometry are expected to require individual manipulation to expose all surfaces to the spray. This is in contrast with liquid penetrants where little manipulation is required and parts can be processed in groups. To achieve the required sensitivity with the current encapsulated penetrant process a nonaqueous wet developer needs to be used. The high cost of this developer is a primary reason for the lack of economic advantage of the present process. However, as was stated in Section 5.5.4 encapsulation manufacturers are evaluating means of eliminating the need for nonaqueous wet developers.

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Presently the encapsulated penetrant process is not a completely dry process as originally conceived. Some capsules do break when they impact the surface of the part, thus releasing oil. Dry removal techniques evaluated were not effective in eliminating background fluorescence, thus a wet removal method needs to be used. Finally, to attain the highest sensitivity, a non-aqueous wet developer is needed. If encapsulated penetrants are to be considered for use on a production basis the problems and disadvantages identified need to be evaluated and resolved. This should primarily involve the effect of additional encapsulating media and capsule characteristics. Different characteristics can be designed into the capsules which may make them more flexible, less susceptible to rupture, easier to remove from the surface, and eliminate the need for nonaqueous wet developers. Additionally, further investigation should be performed on the effect of capsule reconditioning methods on the sensitivity of the process and on the limits of tolerance of the process parameters.

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